PNWD-3264
WTP-RPT-044, Rev 0

Combined Entrained Solids and Sr/TRU Removal from AN-102 Waste Blended with C-104 Sludge Pretreatment Solutions

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March 2003

WTP
Project
Report

Prepared for Bechtel National, Inc.
under Contract 24590-101-TSA-W0000-0004

LEGAL NOTICE

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Test Specification: TSP-W375-01-00003
Test Plan: CHG-TP-41500-019
Test Exceptions: None
R&T Focus Area: Pretreatment
Test Scoping Statement(s): B-39

Battelle—Pacific Northwest Division
Richland, Washington 99352

COMPLETENESS OF TESTING

This report describes the results of work and testing specified by Test Specification TSP-W375-01-0003 and Test Plan CHG-TP-41500-019. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

____________________________________ __________________

Gordon H. Beeman, Manager Date
WTP R&T Support Project

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Summary

The River Protection Project-Waste Treatment Plant baseline for pretreating Envelope C low-activity waste (LAW) at Hanford includes a precipitation step for removing radioactive strontium (Sr-90) and transuranic (TRU) isotopes before the waste is vitrified. The current design basis for the Sr/TRU removal process is the addition of strontium nitrate (0.075M) for isotopic dilution and permanganate (0.05M) for TRU removal at 1M additional sodium hydroxide. Section 5 of the *Research and Technology Plan* (BNI 2002) identifies further research needs, which are illustrated in Figure 5-14, Precipitation Test Matrix.

One need shown in this matrix is optimization of the Sr/TRU precipitation reaction conditions [SOW Ref.: Sec. C.6 Std.2 (a)(3)(ii)(B) and WBS No.: 1.2.10.03 and .05]. The optimization of the Sr/TRU precipitation process and the impact of recycle streams are addressed in Scoping Statement B-39, which is included in Appendix C of the *Research and Technology Plan*. In accordance with Scoping Statement B-39, Test Specification TSP-W375-01-00003, and Test Plan CHG-TP-41500-019, studies were conducted to determine if low levels of reagent provide adequate decontamination conditions for integrated process testing with a mixture of Tank AN-102 waste and high-level waste (HLW) sludge pretreatment streams (supernatant, wash, caustic leach, and rinse solutions from HLW pretreatment of Tank C-104 wastes). The mixture is referred to as "AN-102/C-104 waste blend." Studies with the AN-102/C-104 waste blend include demonstrating that reduced levels of strontium and permanganate addition provide decontamination of the liquid waste (supernatant) to meet LAW requirements for vitrification.

The success criteria for this study include demonstrating that the treated liquid waste meets Specification 2 of the Bechtel National, Inc., contract for removing Sr-90 and TRU elements from the LAW solution; i.e., 20 Ci/m^3 for Sr-90 and 100 nCi/g for TRU (DOE 2000). Blending the AN-102 waste with the sludge pretreatment solutions resulted in over 60% dilution in the Sr-90 and Am-241 activity relative to the sodium concentration. As a result of the waste blending, decontamination factors (DFs) of approximately 5 for Sr-90 (80% removal) and 1.6 for TRU (38% removal) are required to meet a target of 50% below the LAW contract requirements (20 Ci/m^3 and 100 nCi/g, respectively). Since over 90% of the TRU in the AN-102/C-104 waste blend is Am-241, a target DF of 1.6 was established for Am-241.

The objective of the work reported here was to treat a 1-L batch of AN-102/C-104 waste blend and conduct filtration tests to demonstrate that the blending does not affect decontamination and solids removal by crossflow filtration. The 1-L batch of AN-102/C-104 was treated at the optimized process conditions (Hallen et al. 2002a). The treatment conditions provided adequate Sr/TRU decontamination of the AN-102/C-104 waste blend to meet the LAW contract requirements. Multiple samples were analyzed during the treatment and digest of the precipitate. The ratio of Sr-90 to total strontium remained constant during the process testing, and the ratio in the liquid and solids was equivalent. The results showed that strontium exchange/isotopic dilution was nearly complete after 18 minutes of mixing. The Sr-90 DFs showed some variation with time and temperature, as did the total soluble strontium. The decreased strontium solubility with increased temperature has been discussed earlier (Hallen et al. 2002a), but the work described here reflects the first time a detailed study was undertaken and the significant time effect for strontium solubility shown. These results suggest that the Sr/TRU precipitate should be digested for more than 4 hours before filtration; digestion for as long as 24-hour should be considered.

Very little TRU removal occurred without addition of permanganate. The TRU removal showed very little effect of time or temperature once the permanganate was added. Similar to Sr-90 removal, the TRU

removal by permanganate addition was complete 18 minutes after the start of reagent addition. The TRU removal results were similar to earlier small-scale tests (Hallen et al. 2002b) and exceeded the requirements for immobilized low-activity waste (ILAW). The permanganate addition also increased the Sr-90 decontamination by approximately 10%. In addition to TRU removal, permanganate treatment also removed Eu-154, a gamma emitter, which will help reduce the dose in the LAW vitrification facility.

Crossflow filtration tests were conducted with approximately 900 mL of 1.4 wt% solids from Sr/TRU precipitation of the AN-102/C-104 waste blend. A matrix of thirteen 1-hour filtration conditions was tested to evaluate the filterability of the precipitated slurry using a 2-ft-long, single-element, 0.1-µm sintered metal Mott filter in a filtration system installed in a hot cell. The filterability of the slurry was evaluated by varying the transmembrane pressure (20 to 60 psid) and axial velocity (7 to 15 ft/s) in the test matrix, with the permeate being recirculated. The test conditions produced permeate flux that ranged from 0.027 to 0.053 gpm/ft^2. The flux declined linearly with time over the test period. The flux was dependent, to a lesser degree, on axial velocity and transmembrane pressure.

Blending the C-104 HLW sludge pretreatment streams with AN-102 waste had no significant impact on decontamination of Sr-90, decontamination of TRU, or on removal of the combined entrained solids and Sr/TRU precipitate by crossflow filtration. The permeate showed no signs of post-filtration precipitation, indicative of the low concentration of manganese and iron in the final permeate.

Testing began in May 2001, and analytical continued through March 2002. Battelle—Pacific Northwest Division (PNWD) implemented the RPP-WTP quality requirements by performing work in accordance with the quality assurance project plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization. PNWD addressed verification activities by conducting an Independent Technical Review of the final data report in accordance with procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and the reported work satisfied the Test Plan objectives.

References

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1.0 Introduction

This report summarizes work performed by Battelle—Pacific Northwest Division (PNWD) in support of the River Protection Project-Waste Treatment Plant (RPP-WTP) at Hanford. Before the liquid (supernatant) fraction of Envelope C[(a)] wastes (e.g., Tank AN-107 and Tank AN-102 waste) can be disposed of as low-activity waste (LAW), pretreatment is required to remove radioactive strontium (Sr-90) and transuranic (TRU) elements in addition to Cs-137, Tc-99, and the entrained solids. The Sr-90 removal process consists of isotopic dilution and precipitation of $SrCO_3$ by nonradioactive $Sr(NO_3)_2$ addition, and the TRU removal process involves complexant oxidation and subsequent TRU precipitation by permanganate addition. This decontamination method is based on work conducted at Pacific Northwest National Laboratory (PNNL) by Orth et al. (1995). Entrained solids and Sr/TRU precipitate are to be removed via crossflow filtration; Cs-137 and Tc-99 are to be removed by ion exchange. In previous work for the RPP-WTP contractor, PNWD and the Savannah River Technology Center (SRTC) demonstrated a combined entrained solids and Sr/TRU removal process with actual waste samples from Envelope C (Hallen et al. 2000a,b; 2002a,b; Nash et al. 2000a,b).

The WTP proposed process flowsheet for Envelope C waste includes the potential for mixing the incoming tank waste with recycle streams and high-level waste (HLW) sludge pretreatment streams (supernatant, wash, caustic leach, and rinse solutions). These streams would be blended and processed through the Sr/TRU removal process. Optimized treatment conditions were identified in tests with AN-102 waste samples (Hallen et al. 2002a), and were used for small-scale (20 mL) process verification testing on a specified mixture of AN-102 waste samples and C-104 sludge pretreatment solutions, referred to as the "AN-102/C-104 waste blend" (Hallen et al. 2002b). These tests verified that optimized process conditions, which minimized reagent addition and reduced the process temperature to ambient temperature (25 ± 5°C), provided adequate Sr-90 and TRU removal to meet immobilized low-activity waste (ILAW) requirements.

The objective of the work reported here was to treat a larger, 1-L, batch of AN-102/C-104 waste blend and conduct filtration tests to demonstrate that the blending does not affect decontamination and solids removal by crossflow filtration. Treatment and digest were conducted at ambient hot cell temperature (approximately 31°C) with $Sr(NO_3)_2$ addition at 0.02M followed by permanganate addition at 0.02M. The precipitate was digested for 4 hours, and samples removed at various times. Supernatant decontamination data were obtained from the test data. The Sr-90 and TRU decontamination factors (DFs)[(b)] were compared to determine the efficiency of the Sr/TRU removal process.

The treated waste was transferred to a cells unit filter (CUF), and a matrix of filtration tests was completed to determine the permeate flux as a function of process conditions. The filtration results were statistically analyzed to determine the significance of the various process conditions. A filtration model was used to evaluate the components of filtration resistance.

(a) Envelope designations are explained in DOE (2000).
(b) The decontamination factor is defined as the amount of the contaminant in the waste before treatment divided by the amount present after treatment.

The results from Sr/TRU removal treatment and filtration tests are presented in this report. Test conditions and experimental procedures are described in Section 2.0. Experimental results from the tests are discussed in Section 3.0. The major conclusions and recommendations are given in Section 4.0. The appendices contain the test instruction, data sheets, logbook entries, and analytical data.

2.0 Test Conditions and Experimental Procedures

This section describes the conditions used for the Sr/TRU removal and filtration testing, as well as the procedures used for the experiments and analyses.

2.1 Description of AN-102/C-104 Waste Blend

PNWD received 27 bottles of tank waste from Hanford's 222-S Laboratory. The waste material was taken by grab sampling of Tank AN-102 from riser 022 during the period August 7 through 11, 2000, and shipped to the 222-S Laboratory in Hanford's 200 West Area. The sample material was transferred to 500-mL bottles before being shipped to the Radiological Processing Laboratory (RPL) in the 300 Area, where the bottles were inspected upon receipt (Hallen et al. 2002a, Appendix A). All of the samples contained a settled layer of light brown solids with a dark brownish/black standing liquid. Eight of the samples were designated for process testing. The eight as-received AN-102 samples were homogenized to form a slurry and characterized (Urie et al. 2002a). Supernatant and solids from jars of homogenized AN-102 waste were mixed to make a waste material consisting of 2 wt% undissolved solids. Supernatant, wash, caustic leach, and rinse solutions from Tank C-104 processing (Brooks et al. 2000b) were blended with the homogenized AN-102 waste to produce the AN-102/C-104 waste blend. The AN-102 and C-104 materials were blended by Urie et al. (2002b) in accordance with test specification TSP-W375-00-00007 (Johnson 2000). No visible gel formation or net solids increase occurred during the blending process.

A sample of the AN-102/C-104 waste blend was characterized, and the sodium concentration was determined to be 3.2M (Urie et al. 2002b). The target concentration for feed to the Sr/TRU removal process was given as 5.5 ± 0.5M sodium in the test specification, TSP-W375-01-00003 (Reynolds 2001). The waste was evaporated at 50°C until the sodium concentration was estimated to be 5.5M. Samples of the evaporated waste were taken and analyzed before the tests began (Lumetta et al. 2002). The sodium concentration was within the test specifications, 5.5 ± 0.5M after evaporation. The evaporated waste blend was used as feed for Sr/TRU removal and "active" (i.e., with actual waste blend) filtration tests.

Additional filtration tests ("inactive") were conducted with a supernatant simulant that had been treated under similar conditions as the actual waste blend. The treated simulant was used to examine differences in filter types and conditions that could not be obtained in the active filtration tests. The supernatant composition of the AN-102/C-104 waste blend was approximated by mathematically combining composition data from the characterization of the initial AN-102 waste (Hay et al. 2000, Urie et al. 2002a) and the fractions of the various C-104 sludge pretreatments solutions (Brooks et al. 2000b). The sodium concentration was adjusted mathematically to 5.5M, and the resulting composition was used by Geeting et al. (2002) to prepare a supernatant for the inactive tests. The simulated supernatant was treated with 0.02M $Sr(NO_3)_2$, 0.02M $NaMnO_4$, and digested for 4 hours at 25 ± 5°C, analogous to the actual waste blend.

2.2 Development of Test Conditions

The RPP-WTP contract (DOE 2000) requires that the ILAW product contain less than 100 nCi/g TRU and that the average Sr-90 be less than 20 Ci/m^3. Supernatant from Envelope C waste contains levels of Sr-90 and TRU too high to meet ILAW requirements. At the design basis waste sodium oxide loading of

15 wt% waste Na_2O for Envelope C tanks, waste from AN-102 needs DFs of approximately 10 for Sr-90 (90% removal) and 2 for TRU (50% removal) to meet a target of 50% below the ILAW disposal requirements. Blending the AN-102 waste with the C-104 sludge pretreatment solutions resulted in greater than a 60% dilution in the Sr-90 and Am-241 activity relative to the waste sodium concentration. As a result of waste blending, DFs of approximately 5 for Sr-90 (80% removal) and 1.6 for TRU (38% removal) are necessary to meet a target of 50% below the ILAW disposal requirements. Total alpha was measured for all samples and provides a more conservative estimate of the total TRU concentration in the treated samples than Am-241 analysis alone.

Sr/TRU removal process conditions were defined using the results from the earlier optimization studies with AN-102 diluted waste (Hallen et al. 2002a) and small-scale (20-mL) verification tests with the AN-102/C-104 waste blend (Hallen et al. 2002b). Based on these studies, the treatment temperature was specified as 25 ± 5°C, and both strontium and permanganate were added at 0.02M. The target concentrations were based on the final volume after addition of both reagents. No additional hydroxide was specified for this test, since the free hydroxide (0.33M) was shown to be adequate for Sr/TRU removal in previous testing (Hallen et al. 2002a,b). The quantity of each reagent to add to the waste to achieve these values, as well as the actual quantities that were used, can be found in the test instruction included in Appendix A of this report.

The precipitation and filtration testing discussed in this report was conducted in the CUF (see Section 2.3) in the High Level Radiochemistry Facility (HLRF) hot cells (in the RPL). On the day of this test, the ambient temperature in the HLRF A-hot cell was 31°C, whereas during the small-scale tests in the Shielded Analytical Laboratory (SAL) hot cell (also in RPL), the ambient temperature had been 26°C. No provision had been made in the HLRF for chilling the large beaker of waste during the precipitation and digest process; consequently, the precipitation and digest test was conducted at 31°C, slightly higher than the test specification temperature of 25 ± 5°C. The precipitate digest time was specified as 4 hours.

Normally, a clean water flux is specified before and after any waste testing to determine the filter resistance and change with processing. However, the clean water flux test is very sensitive to how well the CUF can be cleaned, and in the hot cell it is difficult to fully clean the CUF after tank samples have been processed. This was also noted in earlier filtration tests (e.g., Hallen et al. 2000b). To remedy this situation for all filtration tests, a standard slurry consisting of precipitated $SrCO_3$ was developed to use for evaluating filter resistance. The $SrCO_3$ slurry was prepared by precipitation of 0.35M $Sr(NO_3)_2$ with excess Na_2CO_3 in a mixture of sodium nitrate and sodium hydroxide. The filter flux of this slurry is less sensitive to the cleanliness of the CUF. Tests were conducted with the $SrCO_3$ slurry in the "cold CUF" (i.e., outside the hot cell) after completion of the hot cell tests to evaluate filter performance and establish reference data for future filtration tests. Table 2.1 shows the targeted filtration conditions that were used for clean water flux and $SrCO_3$ slurry flux testing.

The filtration test conditions (test matrix) were developed jointly by the Contractor, PNWD, and SRTC. The matrix of filtration conditions for the active and inactive testing with the Sr/TRU treated waste and simulant samples is shown in Figure 2.1. The matrix includes 13 combinations (denoted as conditions 1 through 13) of transmembrane pressure (TMP) and crossflow (axial) velocity, each run for 1 hour in the order listed. The filter was backpulsed with permeate between each 1-hour filtration test. The initial filtration conditions (center point of the matrix) were repeated for 3 consecutive hours before filtration conditions were varied. The center point was then repeated in the middle and at the end of testing to assess the effect of filter fouling with time over the course of testing. The permeate flux results from this testing are given in Section 3.0.

Table 2.1. Test Conditions for Clean Water and $SrCO_3$ Slurry Flux Measurements

Test	TMP (psid)	Axial Velocity (ft/s)	Comments
Clean Water Flux with 0.01M NaOH	10, 20, 30[(a)]	11	Hold each condition for 20 minutes; backpulse between conditions
0.35M $SrCO_3$ Slurry	10, 20, 30[(a)]	11	Hold each condition for 20 minutes; backpulse between conditions
(a) If the flux was too high at 30 psi, the pressures were decreased.			

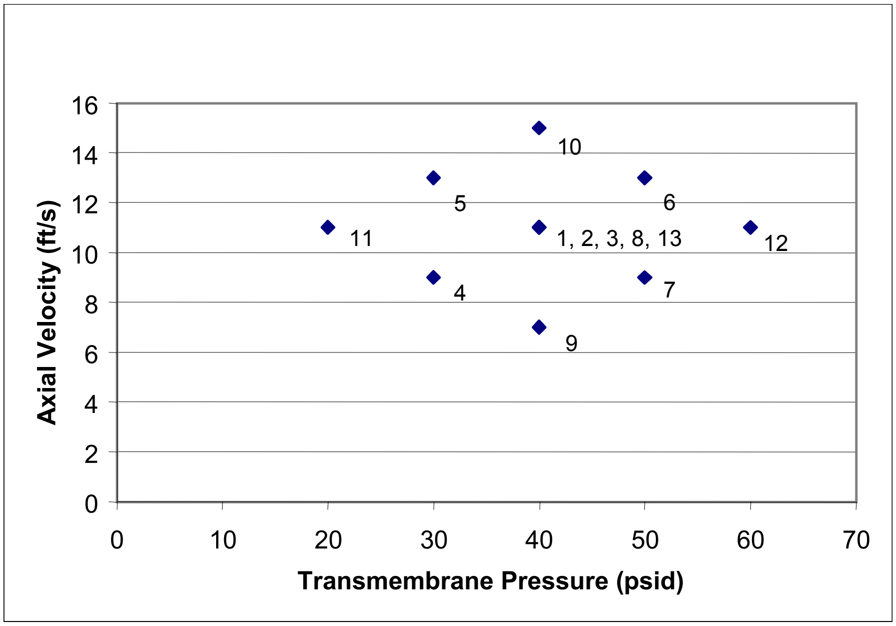

Figure 2.1. Test Matrix of Transmembrane Pressure and Crossflow Axial Velocity Conditions for Filtration of the Sr/TRU Treated Samples (Conditions 1-13)

2.3 Testing Apparatus and Procedure

The precipitation reaction was conducted in a 4-L stainless steel beaker, and stirring was provided by an overhead stir motor with impeller. As noted in Section 2.2, the precipitation and digest were conducted at ambient hot cell temperature (31°C). After the solids digest was complete, the precipitated waste was transferred to the slurry reservoir of the CUF for filtration testing. A new CUF (MOD3) was fabricated just prior to this testing. Modifications and the schematics of the CUF (MOD3) are discussed in Geeting et al. (2002).

During filtration testing, the slurry temperature in the CUF was maintained at the specified value, 25 ± 5°C, by flowing cooling water from an external chiller through a heat exchanger on the feed inlet to the filter. The slurry temperature was measured by a thermocouple installed in the slurry reservoir and controlled by a proportional-integral-derivative (PID) temperature controller that was built into the external chiller. All thermocouples and measuring equipment were calibrated, and the calibration data recorded in the laboratory record book or test instruction.

2.3.1 Filter Element for Testing

For pretreatment testing, a 2-ft-long, 0.1-µm, sintered metal Mott filter was used (as specified by the Contractor). The element is a tube-in-shell design, with the 3/8-inch-ID sintered metal tube mounted inside a 3/4-inch stainless steel pipe with threaded and compression fittings. The inner sintered metal tube (filter) is welded to the outside shell to prevent leaks from occurring at the threaded fitting while testing in the hot cell. Once welded, the filters must be leaked checked and tested before being installed into the hot cell CUF system.

The filters for testing are provided by Mott, which manufactures a full line of sintered metal filters with a wide range of particle size rating (nominal) and for different service applications (gas or liquid). Mott manufactures both an "Industrial Grade" and a "High Purity" (also known as 9-log reduction media) liquid service filter of 0.1-µm rating. The densities of the two filters are about the same (i.e., they both have approximately 30% open pores), with the high purity filter having a larger number of finer pores. While the earlier filtration tests had used the Mott 0.1-µm industrial grade filters, the work discussed here also reports data for the Mott 0.1-µm high purity filters. PNWD received three high purity liquid service filters from Mott, one of which had been installed in the hot CUF for the active filtration tests before the difference became apparent. As a result of conducting the active filtration tests with the high purity filter, a series of inactive tests were conducted, using the cold CUF, with both types of filters. Of the three high purity filters that were sent to PNWD, one had a leak and was not used; one was used for the active testing; and one was used for the inactive testing in comparison with the industrial grade filter. Sections 2.3.2 and 2.3.3 describe similarities and differences in how these filters were evaluated during acceptance testing.

Acceptance testing is conducted with new filters in the cold CUF to verify the filter integrity and condition the new filter so that the initial flux measurements are not artificially high. During acceptance testing, the clean water flux is also measured and compared with previous values to make sure that the filter resistance is not unusually low or high, which could indicate a filter defect. The accepted filter is then available for testing of desired treated sample, in accordance with the conditions outlined in a test instruction.

2.3.2 Acceptance Testing of High Purity Filter Used for Active Testing (Before Hot Cell Installation)

The filter used for the AN-102/C-104 active testing had a 2-ft active length, 3/8-inch ID bore, and 1/16-inch wall thickness. The MOD3 CUF system and filter were first cleaned using tri-sodium phosphate (TSP) solution made from pre-filtered deionized (DI) water. The TSP solution was circulated through the CUF, including the backpulse chamber and all drain lines. The system was then rinsed three times with pre-filtered DI water. After the system was cleaned, shakedown testing was performed on the CUF with a slurry of 5 wt% kaolin clay in DI water. During the shakedown tests, the permeate was observed to contain wisps of color that appeared to be particulate matter. When the filter housing was drained, clear evidence was found that the filter had allowed particulates to pass through. Approximately 90 g of permeate were collected from the filter housing drain. This permeate was dried in an oven, and the total solids content was determined to be 1.0 g or >1 wt%. Apparently, the permeate velocity in the tube was generally lower than the settling velocity of the particulate; hence the solids collected in the filter annulus and the permeate showed only small signs of solids. The filter was rejected for any further testing. [It should be noted that a filter housing drain (1/4-inch Swagelok drain) was added to the new design of the CUF(MOD3), which greatly aided in determining that the filter was leaking solids. It is recommended that a filter housing drain be installed on all filters to be tested.]

Another filter element, ordered at the same time, was prepared by welding the tube in place. The spare filter was cleaned with TSP, rinsed, and tested with a kaolin slurry, as described earlier. This filter showed no signs of leakage. After all shakedown testing was completed and prior to setting up the new CUF in the hot cell, the system was thoroughly cleaned and the clean water flux was measured.

2.3.3 Acceptance Testing of High Purity and Industrial Grade Filters Used in Inactive Tests

After completion of the AN-102/C-104 active testing, it was proposed that the $SrCO_3$ slurry be used instead of the clean water flux measurement to determine the filter resistance. The filters used for inactive tests (a high purity and an industrial grade) were tested with the $SrCO_3$ slurry. As a result of this testing, these two filters were each initially conditioned with the $SrCO_3$ slurry before filtration tests were conducted with the treated (Sr/TRU removal process) AN-102/C-104 simulant.

2.4 Experimental

Once the evaporation of the AN-102/C-104 waste blend was completed in the HLRF hot cells, the waste was transferred to three 500-mL sample bottles (Lumetta et al. 2002). The waste from these bottles was then transferred to a 4-L stainless steel beaker. Some solids remained in the bottom of the bottles, and small amounts of DI water were used to transfer as much of the solids as possible to the large beaker. Duplicate 10-mL samples were used to determine the density of the waste after evaporation. The average density of the waste was 1.25 g/mL. With the waste stirring, an initial sample of the slurry was removed and filtered with a 0.45-µm disposable syringe filter to determine the starting composition of the waste. Stock solutions of 1M strontium nitrate and 1M sodium permanganate were prepared and transferred into the hot cell. These reagents were slowly poured from the bottles directly into the large beaker while the stirrer was running. Additional samples were taken after strontium reagent addition, after permanganate addition, and during precipitate digest. A total of six samples were taken during precipitation and are listed in Table 2.2. The samples were filtered immediately with a 0.45-µm disposable syringe filter into

Table 2.2. Sample Matrix for Precipitation of AN-102/C-104 Waste Blend

Process Step	Sample ID #	Target [Sr]	Target $[MnO_4^-]$	Time from Start of Sr Addition (hr:mm)	Comment
Initial Waste	LS-01	None	None	0:0	Initial waste
Sr Addition	LS-02	0.02M	None	0:18	Sample taken 18 minutes after start of Sr addition, added in 6 minutes
Mn Addition	LS-03	0.02M	0.02M	0:42	Sample taken 18 minutes after start of Mn addition, added in 8 minutes
Digest	LS-04	0.02M	0.02M	1:29	Sample taken after 1 hour of digest
Digest	LS-05	0.02M	0.02 M	2:29	Sample taken after 2 hours of digest

Digest	LS-06	0.02M	0.02 M	4:34	Sampled at completion of digest, 4 hours

the appropriate labeled vial. All samples were collected and transferred to the SAL for analyses. The samples for chemical and radiochemical analyses were acidified and diluted to the appropriate levels for the analytical method. The specific details of the precipitation reaction and observations are given in the test instruction (Appendix A).

After 4 hours of digestion, the waste was transferred from the 4-L beaker to the reservoir of the CUF system. During the CUF testing, five samples of permeate were collected for chemical and radiochemical analyses. These samples (LS-07 through LS-11) are listed in Table 2.3, showing the filtration test number, sample number, time since the start of strontium addition, and lapsed total filtration time.

The volume of waste was insufficient to allow slurry dewatering to be performed in the CUF. As a result, after completion of the CUF testing, the waste was drained, and dead-end filtration was used to dewater the slurry. Two samples of the dead-end permeate (LS-12 and LS-13, Table 2.3) were collected for analyses. The solids collected on the filter were washed with four equal volumes (~30 mL) of 0.01M NaOH. A sample of the composite wash permeate (LS-14, Table 2.3) was analyzed. The washed solids were collected, and duplicate samples dried and digested for chemical and radiochemical analyses (sample LS-16).

Table 2.3. Sample Matrix for Filtration of AN-102/C-104 Waste Blend

Filtration Test No.[(a)]	Sample ID #	Time from Start of Sr Addition	Filtration Time	Comment
1	LS-07	6:16	0:25	First permeate sample from CUF
2	LS-08	7:36	1:45	Sample mid-point of condition 2
3	LS-09	8:38	2:47	Sample mid-point of condition 3
6	LS-10	12:46	6:55	Sample mid-point of condition 6
13	LS-11	20:35	14:44	Sample mid-point of condition 13
Dewater	LS-12/13	55:36	49:45[(b)]	Sampled at completion dead-end filtration
Wash	LS-14	61:03	55:12[(b)]	Sampled at completion of 4 washes

(a) See Figure 2.1 for filtration conditions (axial velocity and TMP).
(b) Total CUF filtration time of 15 hours 8 minutes.

2.5 Chemical Analyses

All of the chemical analyses were conducted at PNWD. The test specification designated the analytes of interest and minimum reportable quantity (Reynolds 2001). Chemical separation and alpha energy analyses (AEA) were performed on selected samples for Am-241. Alpha spectroscopy and total alpha measurements were conducted on all samples. The Sr-90 concentration was determined by chemical separation followed by beta counting. Sodium concentration was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES), as were the other metals listed in the test instruction. Samples were also analyzed by titration to determine the free hydroxide concentration. Along with the standard analyses used for determining process performance, additional analyses were conducted on the permeate and washed solids to support the vitrification testing. The analytical results in support of vitrification are not discussed in detail here, but all of the analytical results are included in Appendix B.

3.0 Results and Discussion

The results of the experiments with the AN-102/C-104 waste blend are discussed here. The Sr/TRU removal process conditions for this test used optimized levels of nonradioactive strontium and permanganate determined in prior tests. A large enough sample was treated to allow filtration testing in the CUF. The results of this testing provide data to demonstrate adequate decontamination of Envelope C tank waste when blended with HLW pretreatment streams (supernatant, wash, caustic leach, and rinse solutions).

3.1 Decontamination of Sr-90 and TRU

Multiple samples were collected during both precipitation and filtration to determine the extent of Sr-90 and TRU decontamination. The samples, as well as sample time and description, are listed in Table 3.1. Duplicate lab analyses were conducted on all samples and the results averaged. The radionuclide composition of the treated samples was compared with the initial composition to determine the extent of

Table 3.1. Sample Identification and Description from AN-102/C-104 Waste Blend

Process Step	Sample ID #	Time from Start of Sr Addition (h:mm)	Time from Start of Filtration Testing (h:mm)	Comment
Initial Waste	LS-01	0:0	--	Initial waste
Sr Addition	LS-02	0:18	--	18 minutes after start of Sr addition, reagent added over 6 minutes
Mn Addition	LS-03	0:42	--	18 minutes after start of Mn addition, reagent added over 8 minutes
Digest	LS-04	1:29	--	1 hour of digest
Digest	LS-05	2:29	--	2 hours of digest
Digest	LS-06	4:34	--	Sampled at completion of digest, 4 hours
CUF 1	LS-07	6:16	0:25	First permeate sample from CUF
CUF 2	LS-08	7:36	1:45	Sample mid-point of condition 2
CUF 3	LS-09	8:38	2:47	Sample mid-point of condition 3
CUF 6	LS-10	12:46	6:55	Sample mid-point of condition 6
CUF 13	LS-11	20:35	14:44	Sample mid-point of condition 13
Dewater	LS-12/13	55:36	49:45[a]	Duplicate samples taken at completion of dead-end filtration
Wash	LS-14	61:03	55:12[a]	Sampled at completion of 4 washes in dead-end filter
Washed Solids	LS-15	61:03	--	Washed solids for energetics and particle size distribution
Washed Solids	LS-16	61:03	--	Washed solids for chemical and radiochemical analyses
(a) Total CUF filtration time of 15:08.				

decontamination. The DF for a specific radionuclide is defined as the concentration of the component in the initial waste divided by the concentration after treatment, corrected by the amount of dilution that occurred during sample treatment:

$$DF = [A]_i / ([A] * MD) \tag{3.1}$$

where $[A]_i$ is the concentration of component A per mass in the initial sample; [A] is the concentration of component A per mass in the treated sample; and MD is the mass dilution, final mass of treated solution divided by the initial mass of solution. The final mass is determined by summing up the mass of initial waste and all dilution, adjustments, and/or reagent additions.

The DFs for Sr-90 and TRU (total alpha) in each sample are shown in Figure 3.1. The Sr-90 DFs were above the target of 5 (see Section 2.2) for all samples; however, the range was quite large from samples LS-6 to LS-12/13. Sample LS-02 was taken after Sr addition, and had very little TRU removed. Adding permanganate (LS-03) increased the removal of both Sr-90 and TRU. The target TRU (total alpha) DF of 1.6 was exceeded when permanganate was added (LS-03 through LS-12/13). The variability in the Sr DFs shows more of a trend with sample number than the TRU DFs. Furthermore, the large drop (~50%) in Sr DF from sample LS-06 to sample LS-07 corresponds to a drop in temperature from 31°C (ambient hot cell temperature) in the precipitation vessel to 23°C in the CUF during filtration testing. Samples LS-07 through LS-11 were all taken during filtration at temperatures of 22-25°C. The final permeate, LS-12/13, was collected by dead-end filtration at ambient hot cell temperature.

3.1.1 Decontamination of Sr-90

A large number of samples were taken over time to examine the rate of isotopic exchange by comparing the ratio of Sr-90 to total Sr in solution. Figure 3.2 shows the ratio of Sr-90 to total Sr with time, and the ratio for the final permeate and solids. The initial waste supernatant has a Sr-90 to total Sr ratio around 20 Ci/g. The initial waste is known to be well under-saturated in Sr and once the addition of $Sr(NO_3)_2$ is complete at 18 minutes, the ratio remains relatively constant at 0.02 Ci/g. The isotopic exchange is

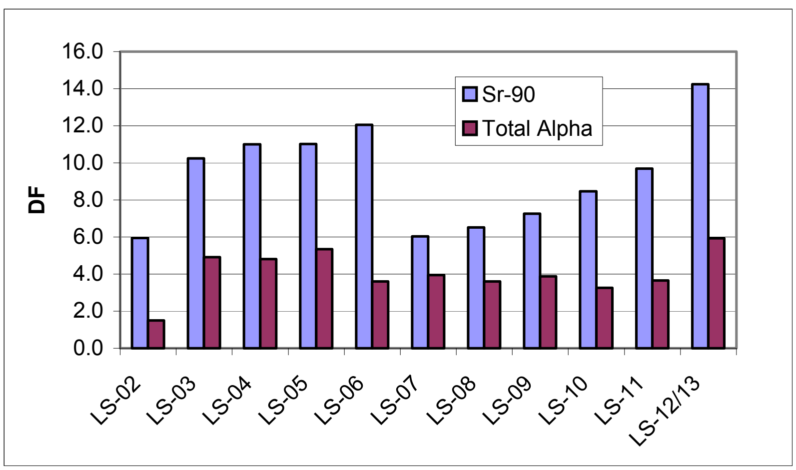

Figure 3.1. Strontium-90 and TRU (Total Alpha) Decontamination Factors for the Samples Taken During Precipitation and Filtration of Treated (0.02M Sr^{+2} and 0.02M MnO_4^-) AN-102/C-104 Waste Blend

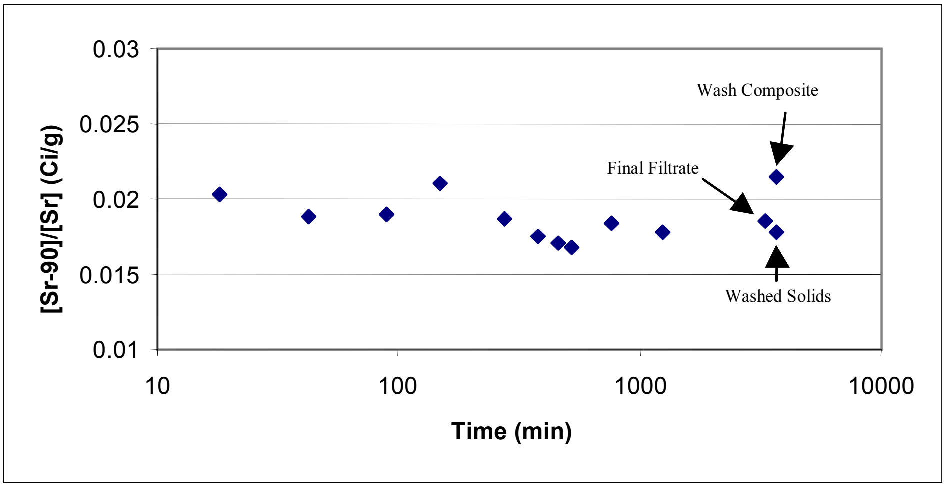

Figure 3.2. Ratio of Sr-90 to Total Sr as a Function of Time Showing Rapid Isotopic Exchange of Sr-90 from Solution

basically complete at 18 minutes of reaction, so the increased decontamination of Sr-90 with time is not a function of increased isotopic exchange. The increased Sr-90 decontamination with time is directly related to the decrease in total Sr concentration, as shown in Figure 3.3. The change in temperature between the precipitate reaction, 31°C, and filtration, 22-25°C, results in a major increase in Sr solubility; however, at both temperatures, additional time results in decreased Sr solubility, which in turn results in increased Sr-90 decontamination with time. The temperature effect on Sr-90 decontamination has been well documented (Hallen et al. 2002a), but this is the first study that has shown the time-dependent Sr-90 decontamination is not a result of isotopic exchange but decreased Sr solubility.

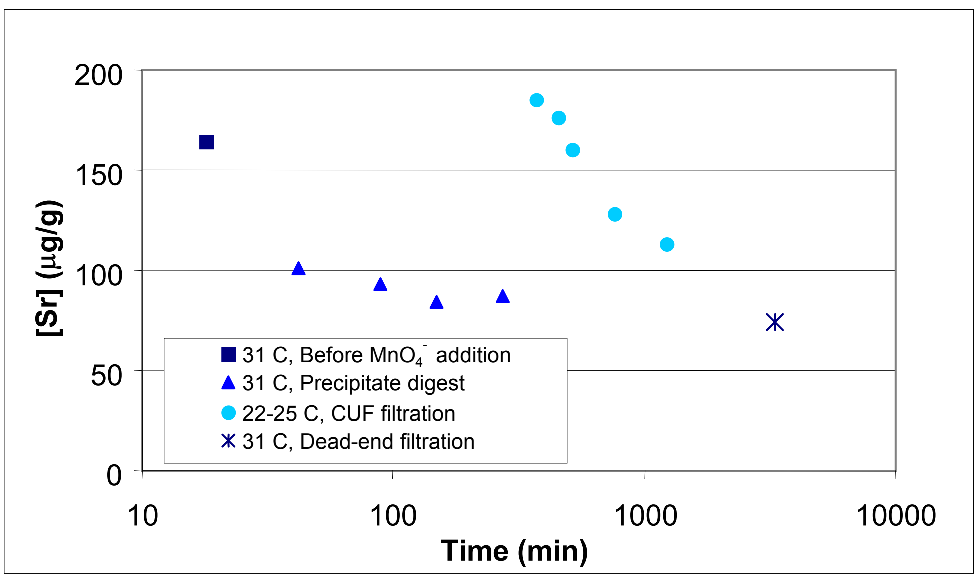

Figure 3.3. Strontium Concentration as a Function of Sample Time and Temperature During Precipitation and Filtration

The Sr-90 DF and total Sr solubility from the earlier small-scale tests (Hallen et al. 2002b) and this study with AN-102/C-104 waste blend are compared in Table 3.2. The data from these tests are consistent considering the sample times, 4 hours versus 18 minutes, and temperatures, 26ºC versus 31ºC, were not the same. The longer reaction time is expected to give better Sr-90 decontamination and lower Sr solubility. Likewise, the higher temperature also is expected to give better Sr-90 decontamination and lower Sr solubility.

Table 3.2. Comparison of Sr-90 DFs and Total Sr Solubility for Treated Samples of AN-102/C-104 Waste Blend

	Small-Scale Tests[a]		**This Study[b]**	
Test Condition	**Sr-90 DF**	**[Sr] μg/g**	**Sr-90 DF**	**[Sr] μg/g**
Sr-Only	6.9	197	5.9	165
Sr + MnO_4^-	8.8	172	10.2	101

(a) 26°C and 4 hours after reagent addition.
(b) 31°C and 18 minutes after reagent addition.

3.1.2 Decontamination of TRU

The TRU decontamination as a function of time and temperature was examined in a manner similar to the Sr-90. Figure 3.4 shows that TRU decontamination was not consistently impacted by time or temperature. The TRU decontamination was significantly greater than the target value of 1.6 once permanganate was added. The permanganate reactions and TRU decontamination were complete when the first sample was removed at 18 minutes of reagent addition and mixing.

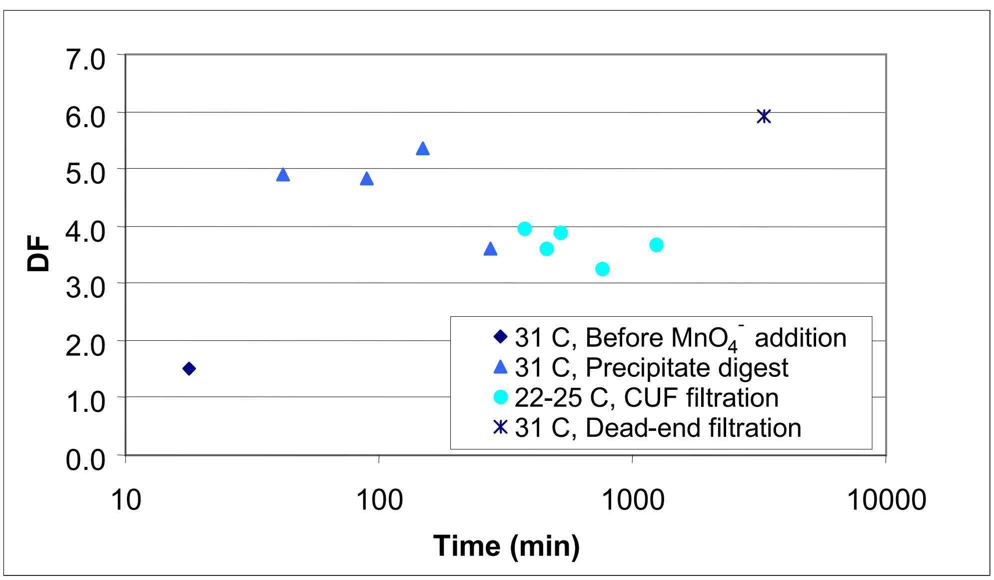

Figure 3.4. TRU Decontamination as a Function of Sampling Time and Temperature

In addition to total alpha counting, the final dead-end filtered permeate was analyzed by additional analytical methods to examine the decontamination of specific TRU elements as well as gamma emitters such as Eu-154. Figure 3.5 shows the DFs for radionuclides that were significantly removed by the Sr/TRU removal process. The TRU is primarily Am-241 (>90%), which is reflected by the similar DF for Total Alpha, Pu-238+Am-241, and Sum of Alpha. Curium is approximately 5% of the remaining TRU and has high DFs. Eu-154 is not a TRU element but a gamma emitter that contributes significantly to the dose rate in the LAW vitrification facility. Removal of Eu reduces dose in the LAW feed and is an added benefit of the Sr/TRU removal process. The other gamma emitters above the background level, Cs-137 and Co-60, were not removed by the Sr/TRU process.

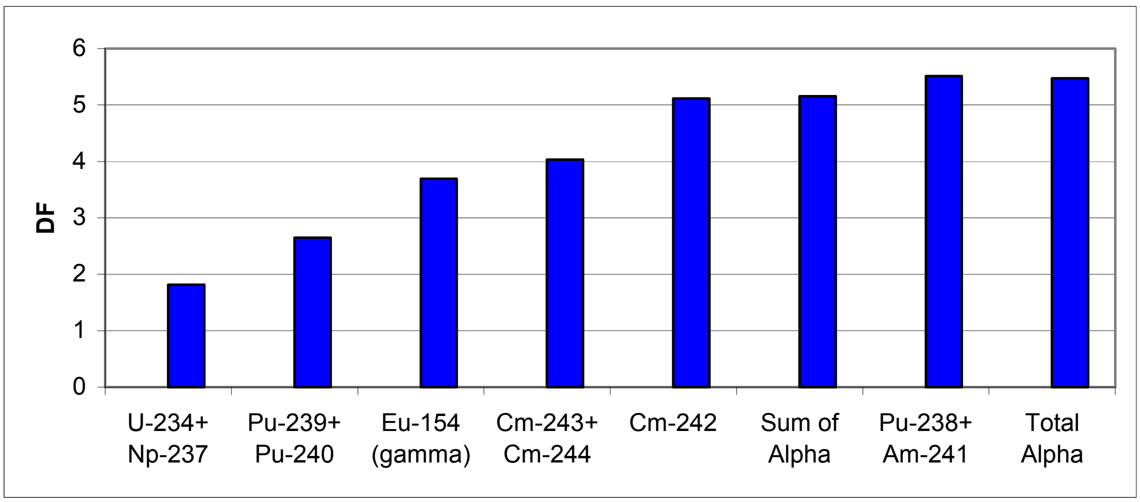

Figure 3.5. Decontamination Factors for TRU Isotopes and Eu-154 in the Final Permeate

The Am-241 and Cm-243+244 DFs for this study and the small-scale tests (Hallen et al. 2002b) with AN-102/C-104 waste blend are compared in Table 3.3. Both studies showed little or no Am and Cm decontamination with Sr addition only. The permanganate addition was required for Am and Cm decontamination, and significantly more decontamination occurred in the large-scale tests discussed here. This additional decontamination was observed when comparing expected DFs from the small-scale, 20-mL, tests to the 1-L bench-scale experiments, and could be a result of differences in reagent addition, including the rate of addition and reagent concentration, or possibly mixing. However, the small-scale tests with the AN-102/C-104 waste blend were lower than expected based on tests with dilute AN-102 (Hallen et al. 2002a), which had DFs of 4.8 for both Am and Cm. Therefore, the results of this study are more consistent with the expected results for AN-102, and the blending does not appear to have impacted the decontamination of TRU elements.

3.2 Chemical Composition

The treated slurry was drained from the CUF system and dewatered by dead-end filtration. The solids were collected on the filter and washed with approximately four equal volumes of 0.01M NaOH. The wash solutions were composited for analyses, and washed solids submitted for moisture analyses and digestion for chemical and radiochemical analyses. The impact of the Sr/TRU process steps on composition of the permeate, the wash composite, and the combined entrained solids/Sr/TRU precipitate is discussed below.

Table 3.3. Comparison of Am-241 and Cm-243+244 DFs for Treated Samples of AN-102/C-104 Waste Blend

Test Condition	Small-scale Tests		This Study	
	Am-241 DF	Cm[(a)] DF	Am[(b)] DF	Cm[(a)] DF
Sr-Only	1.2	1	1.4	0.8
Sr + MnO_4^-	2.7	1.7	4.9	2.9
(a) Cm = Cm-243+244. (b) Am = Am-241+Pu-238.				

3.2.1 Chemical Composition of the Treated Supernatant

Chemical analyses of each sample were conducted using ICP-AES. The ICP data from each sample can be used to determine the impact of reagent addition and sampling time on the chemical composition of the supernatant. However, many key elements were below the detection limit on the instrument (ICP-AES data reports are given in Appendix B). The impact of the Sr/TRU process on the chemical composition of the treated supernatant is best summarized by examining the final supernatant composition and calculating the percent removed relative to the starting waste. Table 3.4 shows the composition of the final permeate sample in µg/g, and the percent change that occurred for the ICP elements. A number of the elements show little or no significant change on treatment: Al, Cd, Cr, K, Na, Ni, and P. It is important that Al, Cr, and P remain in the supernatant so they are vitrified in the LAW melter. The low Cr removal for permanganate treatment is in contrast to the AN-102 diluted waste (Hallen et al. 2002a), which showed significant Cr removal (30 to 50%), but similar Cr removal was found for the small-scale tests with the waste blend (Hallen et al. 2002b). Strontium addition had the most impact on chemical composition; increasing the total Sr from approximately 1 µg/g to 74 µg/g in the treated supernatant. The Sr addition also removed Ca from solution. Significant Fe was removed, which has been noted in all earlier Sr/TRU removal studies. The permeate had low concentrations of Mn and Fe and showed no signs of post-filtration precipitation.

3.2.2 Chemical Composition of the Wash Composite

The primary function of the wash step is to displace the interstitial liquid in the dewatered slurry, but some solids are also dissolved. The treated slurry volume was too low to allow dewatering and solids washing in the CUF. Instead, the dewatered solids were washed on the dead-end filter unit, which is not very representative of washing in the crossflow filter unit, since the solids are not re-slurried with each wash and channeling can occur. Four separate washes were conducted, and the wash permeates were composited for analysis by ICP-AES (Table 3.5). The change in composition of the wash composite relative to the diluted supernatant (permeate) composition gives an understanding of the dissolution or possible precipitation of key elements. A positive percent change indicates the component is greater than expected and likely resulted from dissolution of the solids during washing. Many of the elements were at very low concentration, and the analyses have a higher uncertainty associated with the results.

Table 3.4. Final Permeate Concentration (LS-12/13) and Percent Removal of ICP Metals for the Treated Waste Blend

Analyte	LS-12/13 (µg/g)	Percent Removed
Al	6873	2
Ca	[123]	18
Cd	22	-1
Cr	92	-7
Fe	[3]	54
K	[693]	0
Na	90725	4
Ni	160	0
P	650	1
Pb	[52]	11
Sr	74	(a)
Analyte	**µg/g**	**M**
Free OH^-	2700	0.20
Total Inorganic Carbon(b)	6670	0.69
Total Organic Carbon(b)	9550	0.99

(a) Sr was below the detection limit in starting waste sample.
(b) Determined by the hot persulfate method.
Values in brackets are in low concentration, within 10 times the detection limit, and analytical error is likely to exceed 15%.

Table 3.5. ICP Results for Major Components in Composite Wash Permeate

Analyte	µg/g	Percent Change
Al	1180	--
Ca	[34]	35
Cd	3.96	--
Cr	15.8	--
Fe	[1.3]	55
K	[130]	--
Na	22900	30
Ni	27.8	--
P	99.3	-15
Pb	[13]	30
Sr	15.2	15
Mo	[5]	30
Zn	[2.2]	55

Values in brackets are in low concentration, within 10 times the detection limit, and analytical error is likely to exceed 15%.

3.2.3 Chemical Composition of the Combined Entrained Solids and Sr/TRU Precipitate

The combined entrained solids and Sr/TRU precipitate were washed on the dead-end filter; approximately 30 grams of wet solids were recovered. The filtered, washed solids were determined to be 48 wt% solids. Using the recovered solids mass and total mass of the slurry in the CUF, the slurry was calculated to be 1.35 wt% insoluble solids. The ICP data were also used to estimate the solids content based on mass balance with added Sr and Mn. Using the ICP data shown in Table 3.6 and the known mass of added Sr and Mn, the total insoluble solids were estimated to be 1.38 wt% in the slurry. The two methods agree quite well, suggesting the slurry during filtration in the CUF was 1.4 wt% solids. The initial entrained solids were calculated to be 0.9 wt% undissolved solids. This is close to the value expected based on the initial AN-102 with 2 wt% undissolved solids and the dilution that occurred with blending of the C-104 streams.

3.3 Estimated Sr-90 and TRU Levels in ILAW Glass

The data from this experiment can be used to estimate the Sr-90 and TRU loadings that would be expected in ILAW glass made from the treated supernatant. Values listed in Table 3.7 are given for the current baseline design waste glass concentration of 15 wt% waste Na_2O in the ILAW. The results show that the treated supernatant from all samples taken was below the contract limits for ILAW glass.

Table 3.6. Chemical Composition of Combined Entrained Solids and Sr/TRU Precipitate (Dried Basis)

Analyte	µg/g-Dry Solids
Al	82300
Ba	149
Ca	3300
Cd	[33]
Ce	[245]
Cr	5270
Fe	8225
La	407
Mg	[130]
Mn	66950
Mo	[54]
Na	197500
Nd	[463]
Ni	225
P	1685
Pb	843
Pd	415
Sr	96650
Ti	[38]
Zn	304
Zr	318
Values in brackets are in low concentration, within 10 times the detection limit, and analytical error is likely to exceed 15%.	

Table 3.7. Sr-90 and TRU ILAW Glass Loading for 15 wt% Waste Na_2O

Sample	Sr-90 (Ci/m^3)	TRU (nCi/g)
ILAW Limits:	**20**	**100**
Initial Waste Blend, LS-01	61	66
Treated Samples (Average)	4-10 (7)	10-21 (16)

Strontium and permanganate addition levels of 0.02M and precipitation and digest at ambient temperature (31°C) were adequate to meet the contract requirement. The TRU loading of the initial waste blend was below the contract limit because of the low TRU content in the C-104 sludge pretreatment stream. However, significant TRU removal/decontamination occurred when permanganate was added. As noted earlier, in addition to TRU removal, permanganate removed other gamma emitters such as Eu-154, which will help reduce the dose of the ILAW waste.

3.4 Filtration Testing

All permeate flux data have been corrected to 25°C by using the following equation;

$$\mathrm{Flux}_{25C} = \mathrm{Flux}_T e^{2500\left(\frac{1}{273+T}-\frac{1}{298}\right)} \tag{3.2}$$

where Flux_{25C} is the corrected permeate flux, and T is the temperature (in °C) when the flux measurement (Flux_T) was taken. Raw data for the permeate flux measurements are included in Appendix B.

3.4.1 Clean Water Flux

Clean water flux measurements are taken before and after all CUF testing. A comparison of clean water flux measurements is given in Figure 3.6 for AN-102/C-104 waste blend (this study) and for AN-102 diluted waste reported by Nash et al. (2000b) before and after waste testing. Nash's measurements were obtained at a slightly higher nominal axial velocity, 12 ft/s (3.7 m/s), than used in this study, which was 11 ft/s (3.4 m/s). However, without any solids present, the flux measurements are expected to be dependent on TMP alone. The filters used in each study were different (high purity vs. industrial grade), but both displayed very similar clean water flux measurements before and after waste testing.

3.4.2 AN-102/C-104 Filtration Test Results

At the completion of the 4-hour precipitate digest in the large beaker, the slurry sample was transferred to the CUF feed reservoir for filtration testing. The slurry was estimated to be 1.4 wt% of entrained and Sr/TRU precipitated solids. Only 950 mL of Sr/TRU precipitated slurry were available for the CUF testing. Volumes less than 1 L adversely affect the pump performance and, as a result, the CUF was not able to reach all the targeted matrix conditions (combinations of high TMP and high axial crossflow velocity). The CUF was able to reach all matrix conditions during shakedown testing with higher volumes of slurry and during subsequent testing of the CUF with other tank waste. When the CUF is run with insufficient volume (i.e., less than 1 L of feed), the pump entrains air and pump performance declines. However, the inactive tests with treated AN-102/C-104 simulant showed that air entrained due to insufficient volume does not materially affect the permeate flux (refer to Section 3.5.2).

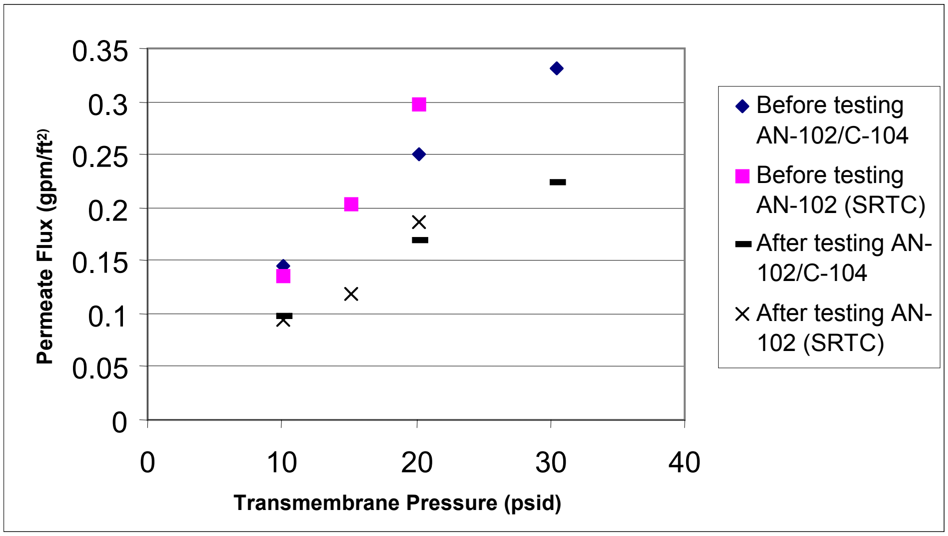

Figure 3.6. Clean Water Flux of the High Purity and Industrial Grade 0.1-μm Mott Filters Before and After Testing Treated AN-102/C-104 and AN-102 Waste Samples

Table 3.8 provides the targeted conditions, average actual condition and the permeate flux, averaged between 10 and 60 minutes after each backpulse. Each condition was run for 1 hour after backpulsing. Note that conditions 1, 2, and 3 have identical targeted flow and pressure. A review of the Nash et al. (2002a,b) filtration data before testing revealed that the majority of the flux decay with time occurred within the first 3 hours of testing. Therefore, the first condition was held for 3 hours to reduce the time dependency of the permeate flux for all subsequent conditions.

Table 3.8. Permeate Flux Data for Targeted and Actual Run Conditions

Condition	Targeted Flow Rate (ft/s)	Targeted TMP (psid)	Average Flow Rate (ft/s)	Average TMP (psid)	Permeate Flux (gpm/ft^2)
1	11	40	11.0	39.9	0.053
2	11	40	11.1	39.9	0.048
3	11	40	10.9	39.9	0.044
4	9	30	9.1	30.8	0.037
5	13	30	12.9	30.8	0.041
6	13	50	9.6	51.0	0.036
7	9	50	(a)	(a)	(a)
8	11	40	10.7	40.7	0.039
9	7	40	7.2	42.9	0.028
10	15	40	10.6	41.7	0.033
11	11	20	10.9	21.3	0.027
12	11	60	8.1	62.7	0.027
13	11	40	11.0	39.6	0.029

(a) Condition 7 was skipped because the targeted conditions for this test were the same as the actual conditions in condition 6.

As expected, and clearly seen from Figure 3.7, the permeate flux has a strong time dependency. As with previous studies (Hallen et al. 2000a,b; Brooks et al. 2000a,b), the flux decreases with time after backpulse and between conditions. These flux values are similar to those reported by Nash et al. (2000b) under similar test conditions. The flux declines with time to values of 0.03 to 0.04 gpm/ft^2, and is only slightly impacted by changing filtration conditions, TMP and axial velocity. The solids loading is quite low in these tests, 1.4 wt%, and because of the low volume of treated waste, <1 L, higher solids loading tests and dewatering in the CUF were not possible.

The average flux data as a function of filtration conditions were analyzed using a statistical software package (SAS Institute, Inc.) to determine the dependency of the flux on time, axial velocity, and TMP. Using linear regression and the three variables, the model predicts 95% of the data variation (Rsquare = 0.95). Of this predictive capability, 89% of the model is influenced by run order (time), 7% by axial velocity, and 4% by pressure. See Figures 3.8 through 3.11 for a graphical presentation of the model. The leverage residuals are the flux variations that remain after applying all the model parameters, except the parameter represented on the y-axis. The leverage plots are shown with confidence curves that indicate whether the test is significant at the 5% level by showing a confidence region for the line of fit. If the confidence region between the curves contains the horizontal line, then the effect is not significant. If the curves cross the line, the effect is significant.

The low flux dependence with pressure indicates that the flux is limited by back-transport of solids away from the membrane surface. Increasing time, decreasing axial velocity, and decreasing pressure all cause the flux to respond lower. The equation for permeate flux as predicted by the model is provided:

$$\text{Flux} = 2.04 \times 10^{-2} - 2.06 \times 10^{-3} \times \text{Time} + 2.13 \times 10^{-3} \times \text{Velocity} + 2.03 \times 10^{-4} \times \text{TMP} \qquad (3.3)$$

With Flux in gpm/ft^2, Time in hours, Velocity in ft/s, and TMP in psid

Using the empirical model described to normalize the flux to remove time dependency (normalized to the first hour of testing), the permeance (flux/pressure) decreases with pressure, with the best-fit curve given Equation (3.4). Figure 3.12 displays this equation graphically.

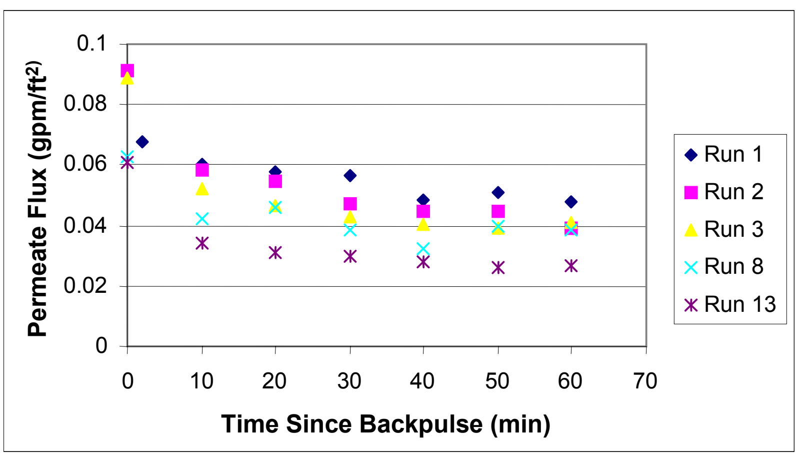

Figure 3.7. Permeate Flux as a Function of Time at Targeted Conditions of 11 ft/s Axial Velocity and 40 psid Transmembrane Pressure (Conditions 1, 2, 3, 8, and 13)

$$\text{Permeance} = 1 \times 10^{-4} + 4.31 \times 10^{-2} \times 1/\text{TMP} \qquad (3.4)$$

With Permeance in gpm/(ft^2 x psi) and TMP in psid

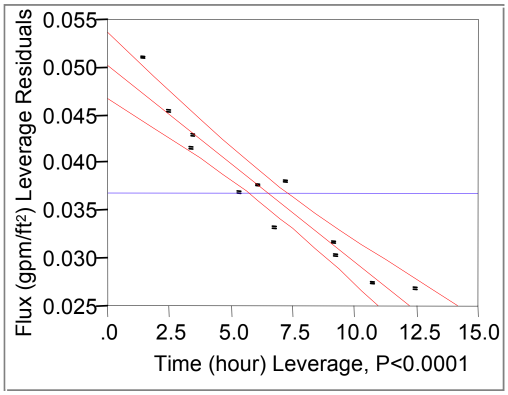

Figure 3.8. Effect of Time on Permeate Flux

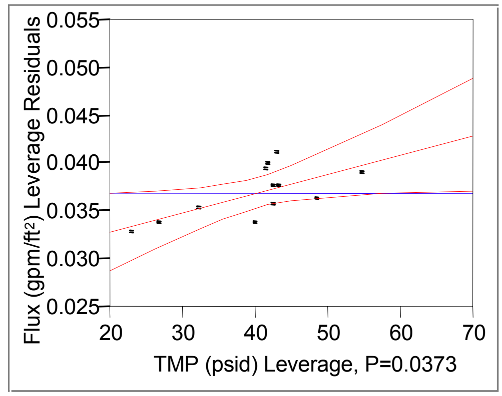

Figure 3.9. Effect of Transmembrane Pressure on Permeate Flux

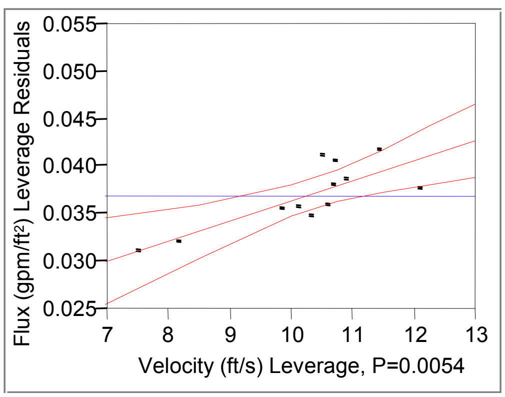

Figure 3.10. Effect of Axial Velocity on Permeate Flux

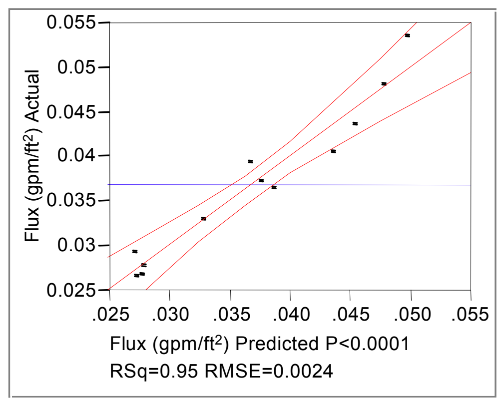

Figure 3.11. Summary of Fit—Whole Model

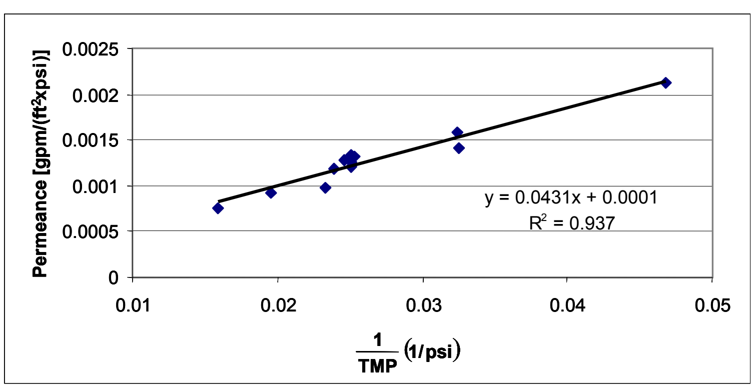

Figure 3.12. Time Normalized (Corrected) Permeance as a Function of TMP

The data indicate that as the pressure goes up, the permeance decreases as the inverse of pressure or, viewed another way, that the flux is weakly dependent on pressure. Increased pressure increases the deposition of particles on the membrane, counteracting the expected result of increase pressure and causing a proportional increase of flux in accordance with Darcy's law (see Equation 3.5). Thus, pressures higher than those tested would be of marginal benefit to the permeate flux.

3.4.3 Investigation into the Cause of the Permeate Flux Decline

It is useful to consider the cause of the decline in permeate flux. First, the hydraulic resistance of the membrane can be determined using Darcy's pressure filtration equation:

$$\mathrm{Flux} := \frac{P}{\mu \cdot R_{total}} \tag{3.5}$$

Where P is pressure, μ is viscosity, and R_{total} is the total hydraulic resistance due to the filter media itself (R_{media}), fouling of the filter media during the test ($R_{fouling}$), and any cake/particulate on the filter surface (R_{cake}), $R_{total}=R_{media}+R_{fouling}+R_{cake}$.

In the clean water flux test, prior to running the treated waste, $R_{fouling}$ and R_{cake} are zero and all the filtration resistance is from the filter media. Any increase in resistance measured during the post-testing clean water flux is attributed to fouling of the filter media. As shown in Table 3.9, the measured 0.1-µm filter media resistance before testing was 8.4 x 10^{11} m^{-1}. After testing, the total resistance increased to 12.4 x 10^{11} m^{-1}; the difference, 4 x 10^{11} m^{-1}, being attributed to fouling that occurred during testing. The total resistance of the filter increased by approximately 50% due to internal fouling during testing in both. It is recognized that the true fouling resistance could be higher, as some of the solids that caused the filter fouling may have dissolved during the CUF cleaning before the final clean water flux was measured. Nevertheless, an increase in hydraulic resistance of this magnitude is generally expected and has been documented for other Hanford tank wastes (Geeting and Reynolds 1997).

Using the resistances determined in Table 3.9, and the average filtration data from Table 3.8, the total hydraulic resistance (R_{total}) during the filtration of the treated waste can be determined, as well as the resistance from solids on the filter (R_{cake}). For this analysis, 3.54 cP was used as the viscosity of the supernatant (the measured viscosity of simulated AN-102/C-104 supernatant). Results of these calculations are shown in Table 3.10.

Initially, during condition 1, R_{media} and R_{foul} accounted for 40% of the total resistance. By the end of the test, even though the sum of R_{media} and R_{foul} increased by 50%, their contribution to the total resistance decreased to 32%, because R_{cake} went up by a factor of 2.

Table 3.9. Filter Resistance Before and After Testing

Filter Resistance	R_{total} (10^{11} m^{-1})	R_{media} (10^{11} m^{-1})	R_{foul} (10^{11} m^{-1})	R_{cake} (10^{11} m^{-1})
Before Testing	8.4	8.4	0	0
After Testing	12.4	8.4	4.4	0

Table 3.10. Filtration Resistance During Testing

Filter Resistance	R_{total} (10^{11} m^{-1})	R_{media} (10^{11} m^{-1}	R_{foul} (10^{11} m^{-1}	R_{cake} (10^{11} m^{-1})
Condition 1	21.6	8.4	0.3[a]	12.9
Condition 13	39.2	8.4	4.4	26.4
(a) A linear increase of the resistance due to filter fouling was assumed.				

Based on this analysis, investigation of the flux decline should be centered on changes in the slurry properties. Figure 3.3 reveals that the concentration of Sr in the permeate was steadily decreasing during the CUF testing. Clearly, the system was not at equilibrium and solids in the slurry were still reacting/changing with run time during the filtration test. This fact alone may account for some of the decline in flux with time.

It is also speculated that the increase in cake resistance may be an artifact of the experimental procedure used for evaluating filtration performance, where a small volume of waste is recycled for many hours while filtration conditions are varied. If the solids in the waste are at all friable, particle attrition will occur with time. Less particle attrition is expected in the plant where the waste is dewatered immediately upon receipt. A comparison of the volumetric flow rate of the retentate (i.e., the slurry) with the volumetric flow rate of the permeate indicates the number of times the waste cycles through the crossflow system. Using the test conditions from the CUF[a] and the proposed plant filter geometry,[b] the calculated number of times the waste would recycle through the plant's filtration system is estimated to be 230. This number is likely higher than expected because the plan for the waste treatment plant is to stop dewatering at ~20 wt% undissolved solids, and the 230-cycle estimate assumes compete conversion (dewatering) to permeate. It is estimated that the slurry was recycled 11,000 times through the filtration system during the CUF testing. When comparing filtration test results for the CUF (~11,000 cycles) with that expected in the plant (<230 cycles), less particle attrition should be expected in the plant.

3.4.4 Particle Size Determination

The particle size of the final washed solids (combined entrained solids and Sr/TRU precipitate) was determined using a Microtrac X-100 particle size analyzer. Particle size data were obtained at two flow velocities and after sonication. The particle size distribution based on volume (Figure 3.13) clearly shows the waste solids form agglomerates that are very sensitive to shear forces, both increased velocity and sonication. The number-basis particle size distribution (Figure 3.14) gives a better indication of mean particle size, which was reduced approximately 40%, from 1.79 µm to 1.13 µm with an increase in solution velocity and sonication. Manganese solids prepared from permanganate treatment of waste simulant were found to be amorphous and sub-micron in particle size by scanning electron microscopy and transmission electron microscopy analyses. The Mn solids form larger agglomerates that are very sensitive to shear forces, as shown in the comparison of the particle size analyses of Mn solids with and without sonication in Figure 3.15. The particle size data help to explain the reduced filter flux.

(a) 0.03 gpm/ft^2 at 11 ft/s axial velocity and 40 psid.
(b) Filter tube ID ½ inch; length 90 inches.

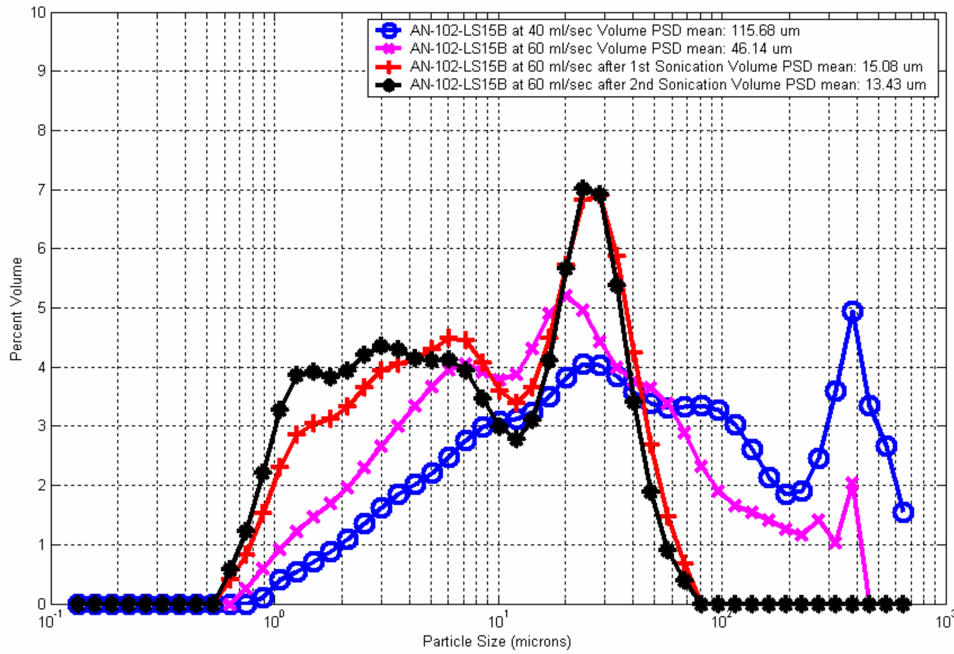

Figure 3.13. Volume Basis Particle Size Distribution of the Washed Solids

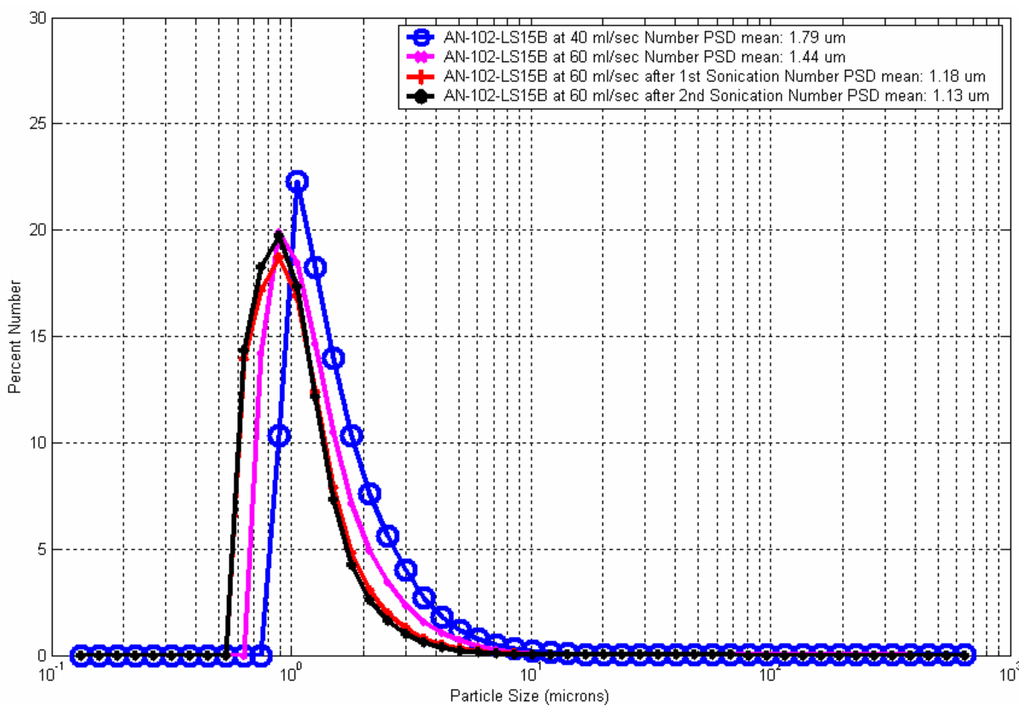

Figure 3.14. Number Basis Particle Size Distribution of the Washed Solids

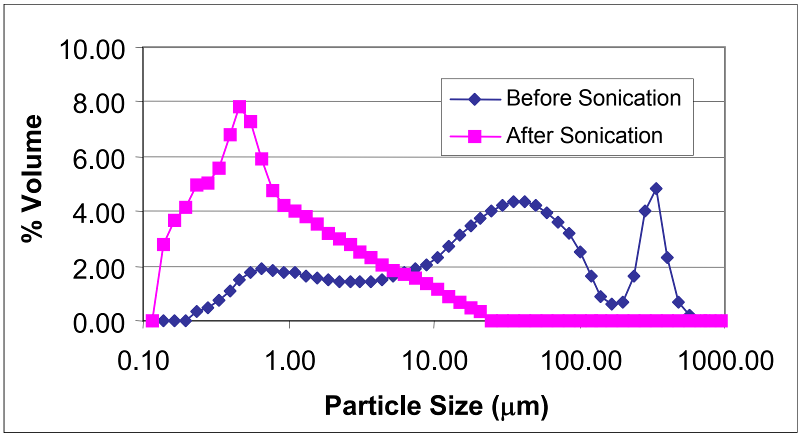

Figure 3.15. Particle Size Analyses of Simulated Mn Precipitate

3.5 Inactive Filtration Test Results

This section describes filtration tests conducted in the cold CUF to provide additional filtration data for comparison purposes. The cold CUF is identical to, and was fabricated at the same time as, the hot CUF installed in the hot cell. Test were conducted with clean water, a $SrCO_3$ slurry, and samples of treated AN-102/C-104 simulant.

3.5.1 Filter Comparison

Filter flux measurements were made to compare the clean water flux from the two filter types (industrial grade and high purity) (Figures 3.16 through 3.19; note, in the figures, industrial grade is designated as IG and high purity as HP). One high purity filter was tested and two industrial grade. All were new and had never been used for filtration testing.

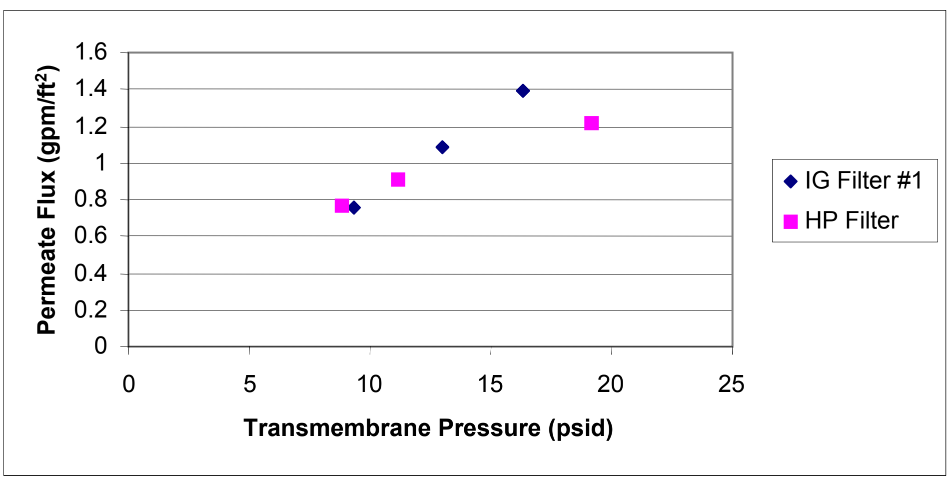

Figure 3.16. Comparison of Clean Water Flux Measured with New 0.1-µm High Purity and Industrial Grade Filters

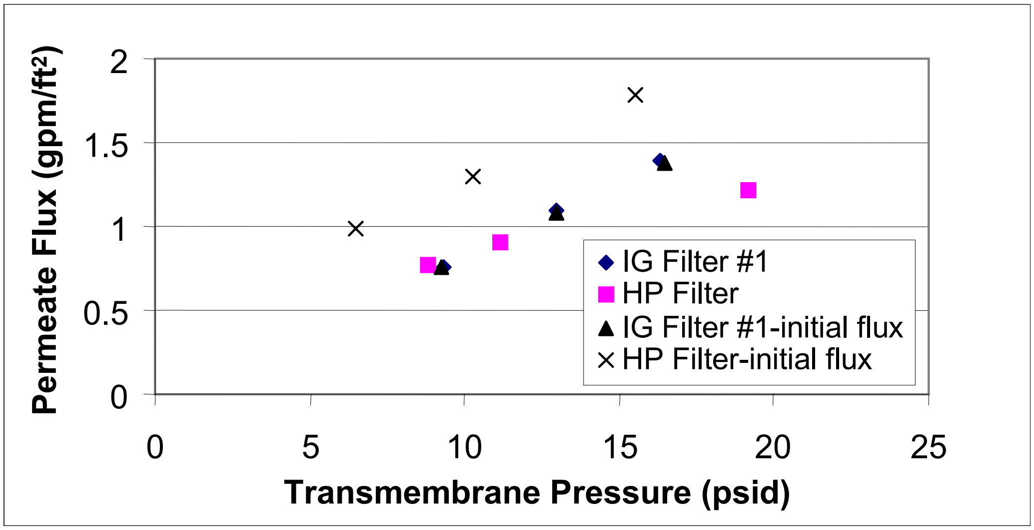

Figure 3.17. Comparison of Clean Water Flux with Initial Flux Data Added

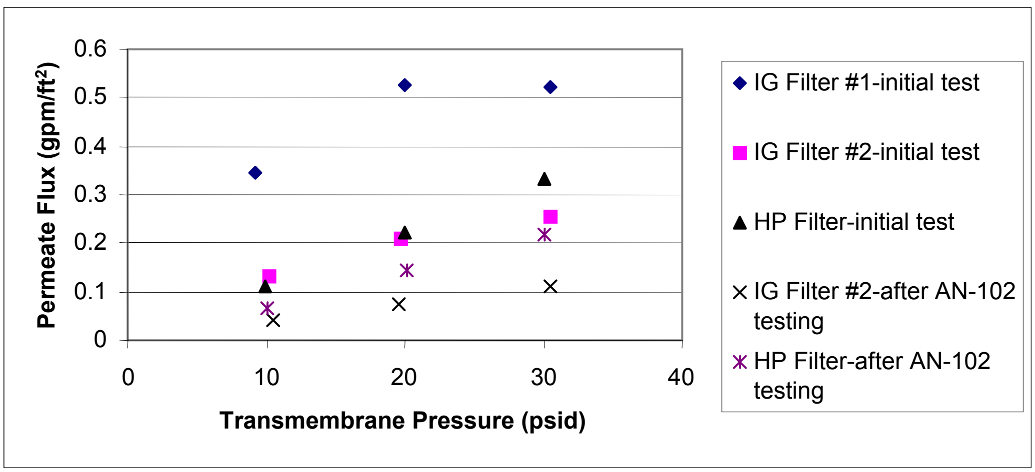

Figure 3.18. Comparison of $SrCO_3$ Slurry Results with the HP and IG Filters

As shown in Figure 3.16, the average permeate flux for IG #1 and HP is very similar. However, a closer evaluation of the time-dependent flux data for these filters indicates that the IG filter had a stable permeate flux with time. In contrast, the HP filter flux was varied immediately after backpulsing. This contrast is most clearly illustrated Figure 3.17, which shows the same data as Figure 3.16, but also includes the data immediately after backpulsing.

If the CUF system was clean and only clean water was being filtered, neither filter should have shown any time dependency. The results in Figures 3.16 and 3.17 were surprising, as the cold CUF was completely disassembled and each part was hand cleaned prior to these tests.

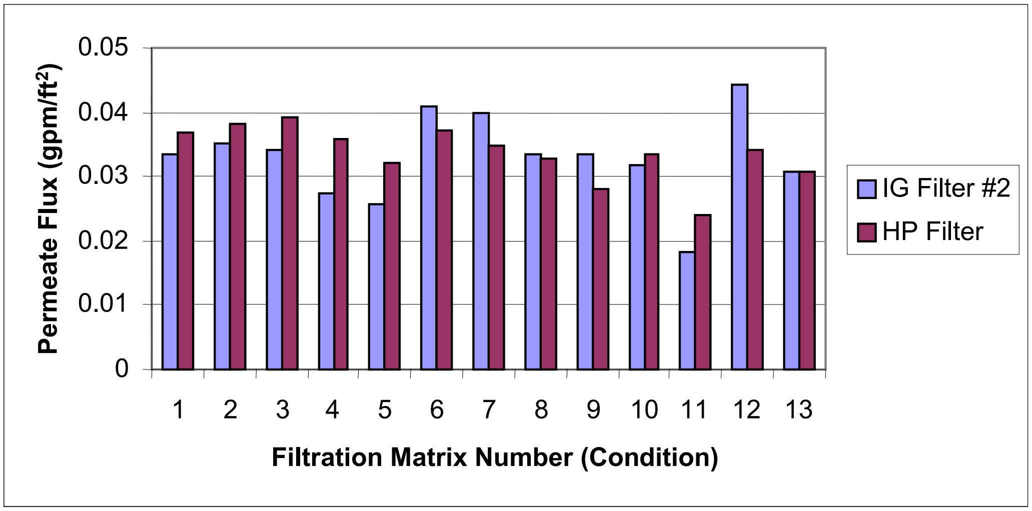

Figure 3.19. Comparison of Filtration Flux for the IG and HP Filters with Treated AN-102/C-104 Simulant

In contrast to the clean water flux results, the filtration data for the $SrCO_3$ slurry, used to further compare filter performance, indicate that IG #1 is initially more permeable than the other filters, as shown in Figure 3.18. (After the initial $SrCO_3$ testing, IG #1 was set aside for filtration testing in the hot cell with actual wastes, and IG #2 was tested.) IG #2 and the HP initially have approximately the same permeability, with the HP filter having a higher measured permeability at 10 psid but lower at 30 psid TMP. A comparison of the two IG filters indicates there was significant variability in the initial permeate flux. Mott indicated that the IG should initially be more permeable than the HP, and these results are generally consistent with that information. It is interesting that after the AN-102/C-104 simulant testing, the IG filter became less permeable than the HP filter, suggesting the IG filter is more susceptible to fouling.

Both types of filters were tested with a treated AN-102/C-104 simulant that contained approximately 0.8 wt% undissolved solids. These results are presented in Figure 3.19. An order-of-magnitude analysis of the hydraulic resistances for filtration indicates that the differences in filter permeability observed has very little influence on the overall permeate flux. This conclusion is supported by the data, as the overall averaged flux measured was within 2.5%, at 0.0329 and 0.0337 gpm/ft^2 for the IG and HP filters, respectively. The IG showed more variability with the standard deviation of 0.00675 versus 0.00424 for the HP. As can be seen in the comparison of the results from the $SrCO_3$ slurry and treated AN-102/C-104 simulant, there is not a significant difference between the IG and HP filters.

3.5.2 Effect of Entrained Air on Filter Flux

To determine the effects of entrained air on filter flux, immediately following the completion of filter test matrix with treated simulant (using the high purity filter), the slurry volume was reduced from 1500 mL to 900 mL. Two conditions were then run at these reduced volume conditions for 1 hour each, 21 psid TMP, 11 ft/s; and 40 psid, 11 ft/s. The data were then used to develop an empirical model using the statistical analyses software program (see Section 3.4.2). The empirical model derived from the simulant data was used to predict the permeate flux measured at the additional processing time for these low-volume test conditions.

The results for the low-volume filter tests could be compared with the model's predicted values to ascertain if there were any significant differences in the observed and predicted flux. Figure 3.20 shows the middle line, a regression line, through the observed simulant flux values that generated the empirical model. The two outer irregular lines are the 95% prediction bands for individual flux values; 95% of simulant runs would be expected to generate observed flux values between two lines. The two plus symbols (first symbol from the left and fourth from left) are the two low-volume observed flux results, and they are well within their corresponding prediction range based on the simulant model. Any values inside the outer confidence lines would have been acceptable, and these values are well within those bounds. Therefore, evidence suggests that the low-volume filtration does not adversely affect the filter flux, but limits the ability to reach the higher TMP and crossflow velocity conditions of the filtration test matrix.

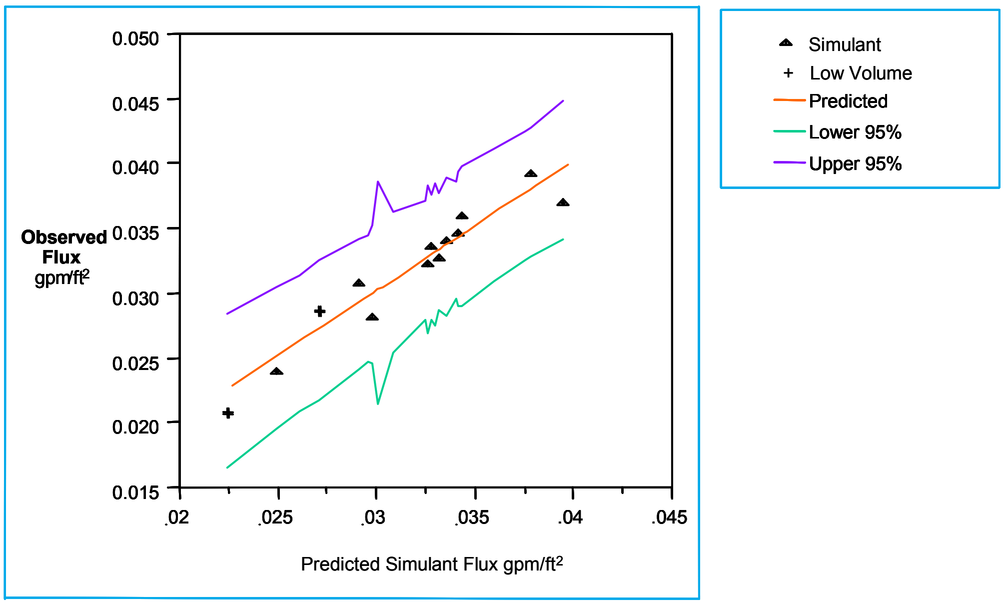

Figure 3.20. Comparison of the Simulant Empirical Model with Low-Volume Simulant Permeate Flux Measurements

4.0 Conclusions and Recommendations

Reagent addition and precipitate aging/digest were conducted on AN-102/C-104 waste blend samples at ambient hot cell temperature (31°C). This temperature was outside the test specification of 25 ± 5°C, and was higher than expected, but no provision had been made for cooling the large, 4-L, beaker before the test started. Reagent addition was $Sr(NO_3)_2$ at 0.02M and $NaMnO_4$ at 0.02M. The waste blend was treated at the initial free hydroxide level, 0.33M. Multiple samples were taken during precipitation, digest, and filtration. The DFs for Sr-90 for all of the samples taken were higher than 5, which is an adequate decontamination to meet ILAW disposal requirements. The addition of permanganate increased the Sr-90 decontamination, and the Sr-90 DFs increased with time. However, upon introduction and simultaneous cooling of the sample in the CUF for filtration testing, the Sr-90 DF decreased as a result of the decreased filtration temperature, 22-25°C. This decrease was expected, as earlier results have shown the Sr-90 DF to increase with increasing temperature. The results also show that precipitation temperature does not determine the DFs of the Sr/TRU process, but filtration temperature does have an impact. These results verify that, for Sr-90 removal, the precipitation temperature and filtration temperature should be the same and the $Sr(NO_3)_2$ addition can be reduced to 0.02M.

The primary mechanism for Sr-90 removal is isotopic dilution with the added nonradioactive $Sr(NO_3)_2$. Examining the Sr-90 to total Sr data ratio for the samples as a function of added reagents and time showed the isotopic exchange was basically complete at 18 minutes. The increased Sr-90 decontamination with time was not a result of increased isotopic exchange but continued precipitation (reduction of total soluble Sr). The permanganate addition also reduced the total Sr levels, which resulted in a higher Sr-90 DF. The reduction in total soluble Sr by permanganate treatment is likely a result of oxidation of the chelating agents, EDTA and HEDTA, and possibly the increase in carbonate concentration.

The TRU decontamination in the AN-102/C-104 waste blend occurred after the permanganate was added. The TRU removal exceeded the requirements for ILAW glass by a factor of 5. The TRU decontamination showed no consistent trend with time or temperature. The TRU decontamination at 18 minutes for the start of permanganate addition was very similar to the final value obtained after 55 hours of additional testing. The TRU removal for this study was higher than from the 20-mL-vial test (Hallen et al. 2002b) and consistent with AN-102 diluted waste (Hallen et al. 2002a). This result suggests that blending had no impact on TRU removal. The initial TRU levels were also significantly decreased with the waste blending, such that the waste without treatment was below the ILAW levels. However, permanganate treatment has the added benefit of reducing gamma levels in the LAW feed, i.e., removal of Eu-154.

Crossflow filtration testing was conducted with the treated waste slurry consisting of approximately 1.4 wt% total insoluble solids, combined entrained solids, and Sr/TRU precipitate. Filtration performance was similar to the unblended AN-102 waste filtration tests conducted at SRTC (Nash et al. 2000b). Long-term filtration performance for the Sr/TRU treated waste is expected to be in the range of 0.02 to 0.04 gpm/ft^2, but will decrease with higher solids loading and increased filter fouling. Additional conclusions include:

- For a slurry of 1.4 wt% insoluble solids, the average permeate flux ranged from 0.027 to 0.053 gpm/ft^2. Statistical modeling indicates that the dominant variable affecting the permeate flux data was time, followed by axial velocity and TMP.

- The bulk of the permeate flux decline with time may be caused by increased hydraulic resistance of the slurry rather than in-depth fouling of the filter. The increase in resistance is believed to be a result of continued solids formation during the run, as the slurry was not at equilibrium. The soluble strontium concentration in the permeate steadily decreased during the run, resulting in an increased undissolved solids loading.

- Another contributing factor to the permeate flux decline is believed to be particle deagglomeration/ attrition due to severe shearing conditions in the CUF. This conclusion is supported by particle size analyses that showed a 40% decrease in mean particle size on sonication of the washed solids, which is theoretically sufficient to cause the permeate flux decline observed.

- Operating volumes less than the minimum (<1 L) result in air entrainment, which degrades the pump performance. It was demonstrated with treated AN-102/C-104 simulant that the air entrainment does not materially affect the resulting permeate flux.

- The comparison of industrial grade and high purity (also known as the 9-log reduction media) 0.1-μm filters indicates the permeate flux results differ as a function of filter conditions for the simulant tested. However, the average permeate flux measured for the treated AN-102/C-104 simulant with both filters was within 2.5%, at 0.033 and 0.034 gpm/ft^2 for the industrial grade and high purity filters, respectively. The permeate flux from the industrial grade filter was influenced more by changes in axial velocity and TMP (made during testing of the standard test matrix), as the standard deviation was 0.0068 gpm/ft^2 (vs. 0.0042 for the high purity filter).

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Appendix A

Test Instruction

Battelle Test Instruction	**Document No.: TI-RPP-WTP-073** **Rev. No.: 0**
Title: Sr/TRU Removal from AN-102: Small Scale Active Tests to Determine Adequate Treatment Conditions	
Work Location: RPL SFO HLRF	
Author: RT Hallen	**Effective Date: New** **Supersedes Date: New**
Use Category Identification: Reference	
Identified Hazards: _x_ Radiological _x_ Hazardous Materials __ Physical Hazards __ Hazardous Environment __ Other:	**Required Reviewers:** _x_ Author _x_ Technical Reviewer __ RPL Manager __ Project Manager __ RPG Quality Engineer __ CHG

Are One-Time Modifications Allowed to this Procedure? _x_ Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? _____ Yes or __x__ No

FOR REVISIONS:
Is retraining to this procedure required? _____Yes __x__ No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
_____Yes _____No __x__ N/A

Approval	**Signature**	**Date**
Author	Richard T. Hallen	5/04/01
Technical Reviewer	John G. H. Geeting	5-4-01

I. Applicability

This test instruction details partial implementation of PNNL Test Plan TP-PNNL-WTP-019, *Sr/TRU Precipitation and Crossflow Filtration Test Plan*. The specific work detailed in this test instruction is the large-scale Sr/TRU precipitation and subsequent crossflow filtration tests in the Cell Unit Filter (CUF). The precipitation work will be conducted according to RPL *Routine Research Operations Procedure*, RPL-OP-001. The filtration testing will be conducted in accordance to the Battelle Operating Procedure, TPR-RPP-WTP-058, Ultrafiltration Testing. These tests will use the integrated process waste, AN-102/C-104. The waste mixture was evaporated in HLRF to a sodium concentration of approximately 5.5M. Approximately 1-L of the evaporated AN-102/C-104 will be available in HLRF hot cells for these tests.

Work is to be performed by hot cell technicians under the supervision of a cognizant scientist/engineer. The cognizant scientist/engineer shall be responsible for implementation and adherence to this test instruction. This instruction is specific to:

- Sr/TRU and entrained solids removal from an integrated envelope C waste; AN-102/C-104 blend
- precipitation of Sr and TRU by strontium nitrate and sodium permanganate reagent addition, and
- crossflow filtration tests performed at Battelle in the RPL, by staff in the Environmental Technology Division.

II. DRD Reference

Task 9.2.2 of the Development Requirements document identifies activities for Active Integrated Pretreatment Testing (i.e., mixing AN-102 and C-104 HLW permeate and subsequent process verification testing).

III. Schedule Reference

The RPP WTP Research and Technology schedule for Y2001 identifies this activity as R20850, RTPT Integration of Pretreatment systems (PNNL). The corresponding activities on the PNNL schedule are 02.05.

IV. Justification

Sr/TRU removal tests with AN-102 at Savannah River last year were successful in demonstrating adequate Sr and TRU decontamination. Subsequent tests at Battelle examined the effect of minimizing reagent addition, and the impact of integration of HLW process streams (C-104 sludge washing/leaching solutions). This tests will use AN-102/C-104 homogenized feed to verify decontamination levels at low levels of reagent addition, and prepare a large enough batch of combined entrained solids and precipitate to conduct crossflow filtration tests in the CUF.

V. Objective

The objectives of this task are to:

- Perform Sr/TRU precipitation tests on a large sample of the AN-102/C-104 waste at low levels of reagent addition (0.02M strontium nitrate, 0.02M sodium permanganate, and no additional hydroxide).
- Collect samples, filter immediately, and analyze the precipitation mixture with time for sodium, total strontium, ^{90}Sr, and total alpha counting to determine decontamination factors.
- Conduct filtration tests in accordance with a test matrix to determine the filterability of the combined entrained solids and Sr/TRU precipitate as a function of time, transmembrane pressure, and crossflow velocity.
- Prepare treated filtrate/supernatant for ion exchange testing.

- Wash solids for analyses and energetics/gas generation testing.
- Evaluate data and report the results.

VI. Success Criteria

The success criteria are to demonstrate that the treated waste meets specification 2 of RFP solicitation DE-RP27-00RV14136 for removal of ^{90}Sr and TRU elements from the LAW solution and the combined entrained solids/precipitate can be filtered by crossflow filtration.

VII. Spill Protection/Response

Hot cell technicians shall conduct tests in a manner to minimize the impact of a spill. In the event of a spill, all practical efforts will be made to recover the test material. Recovered material will be segregated and retained pending a decision by the cognizant scientist/engineer on how to proceed.

VIII. Feed Description

These Sr/TRU removal and filtration tests will use the remaining AN-102 /C-104 homogenized feed. This material was prepared by evaporating the AN-102/C-104 dilute, homogenized sample to approximately 5.5 M sodium.

IX. Equipment Description

The Sr/TRU removal test will be conducted on a large scale, approximately 1-L, in HLRF. A 4-L stainless steel beaker with overhead mechanical stirrer will be used for precipitation. After 4 hours of precipitate digestion, the slurry will be transferred to the CUF resorvior for filtration tests. The CUF is described in Ultrafiltration Testing Operating Procedure.

X. Work Instructions

1.0 Relevance

This test instruction is to be used to perform Sr/TRU removal and crossflow filtration tests with AN-102/C-104 homogenized waste. The homogenized sample of AN-102/C-104 is currently located in HRLF. Homogenization of AN-102 tank waste samples and C-104 wash/leach solutions occurred in HLRF under Test Plan 41500-004 and according to Test Instruction 41500-005. Evaporation was conducted according to Test Instruction PNNL-WTP-050.

2.0 Supporting Documents

This test instruction is to be used with the Routine Research Operations Procedure, RPL-OP-001, Battelle Operating Procedure for Ultrafiltration Testing, TPR-RPP-WTP-058, and the Sr/TRU Removal Test Plan, TI-WTP-019. Sr/TRU removal test conditions and analytical requirements were designated in Test Specification TSP-W375-01-00003 from CHG. These are follow-on studies of work performed at Battelle and SRTC for the RPP-WTP Contractor (BNFL-TI-29953-037, -040, -041. -043, -052, and -063).

3.0 Responsible Staff

The staff responsible for executing this test plan are as follows.

- Task Manager – Rich Hallen
- SFO Manager – Randy Thornhill
- Test Scientists/Engineers – Rich Hallen, John Geeting, Dave Jackson, Lynette Jakoda
- Hot Cell Technician – list names/location/work performed
  Tim Retting – ppt. Steve Halstead – CUF/Dewatering Don Rinehart – C4

- Radiological Control Technician

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

1. Twelve 20 mL glass vials for samples, pre-labeled as follows: LS-01 through LS-12. Record tare weight on each vial before transfer into hot cell.
2. 6 - disposable syringes and 0.45 micron syringe filters. With extras on hand if filtering is difficult.
3. 1-L storage bottle for filtrate product
4. 4-L stainless steel beaker
5. 1-L disposable deadend filtration unit with 0.45 um filter.
6. Long reach disposable pipettes for sampling.
7. Two 10 mL volumetric flasks for determining the density of the initial AN-102/C-104 supernatant.

4.2 Equipment

1. 160 gram and 2 kg capacity analytical balances
2. overhead stir motor and impeller
3. Calculator
4. Clock/Stop-watch
5. Thermometer or temperature reading device

4.3 Reagents Needed In Hot Cell (see prep sheet)

1. 21 mL of 1M $Sr(NO_3)_2$
2. 21 mL of 1M $NaMnO_4$

4.4 Other Supplies

1. Copy of this TI to record data
2. Laboratory Record Book (use red bound lab notebook, record book BNW-13733 ← ppt + solids washing

CUF Log book # 14048 -

5.0 Instructions

The laboratory record book (LRB) shall be used to record observations and other testing information as required by this test instruction. All test conditions shall be recorded on a copy of this test instruction or in the lab notebook.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new supplies and bottles for each sample as much as practical.

Keep lids on containers to minimize the potential for spills and to prevent evaporation.

5.1 Prestart

5.1.1 Prepare strontium and permanganate solutions according to the attached preparation sheet. Calculate solution densities and record these values. All vials should be labeled and tared before they are taken into the hot cell. NOTE: Tare weigh bottle/vials with caps/lids

5.1.2 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Modified materials/equipment as needed for remote handling.

Record Unique ID # of reagents:

1M $Sr(NO_3)_2$ 1M $Sr(NO_3)_2$

1M $NaMnO_4$ 1 M NaMnO4

5.1.3 Review the test conditions in this test instructions. Note the calculation worksheet, which gives quantities of reagents to be added. Materials may be dispensed on a volume basis but always record the weight.

5.1.4 Obtain the following information:

M&TE List: Note Balance Location, cell #

2Kg weight = 2000.01 grams

2 Balance(s) : (record for each balance used)

Calib ID 111319277 Calib Exp Date 2-2002

Location A cell-North 200g = 200.015 grams

1 temperature reading device (thermometer or thermocouple/reader): 111321643 2-2002

Calib ID 2013 Calib Exp Date A cell-south 8/2001

Location A cell south

31°C 2:30 Am 5/7/01

5.2 Sr/TRU Precipitation

Record the initial weight of the empty 4-L beaker. Set up the 4- L beaker and overhead stir assembly.

Weight of empty beaker 768.04 g

Locate sample bottles. TI-050 indicates that the evaporated feed is in three separate bottles labeled AN-102/C-104 FEED#1, AN-102/C-104 FEED #2, and AN-102/C-104 FEED #3. Record the appearance of each bottle of waste. Note color and presence of solids. Additional solids may have formed with time. Record weights, and quantities transferred to the large stainless steel breaker. Use approximately 25 mL to wash/rinse each bottle. Record the amount of water transferred to large beaker.

AN-102/C-104 FEED#1 705.39 grams very little solids - gray color Light brown

Full Bottle weight 705.39 Empty Bottle Weight 320.24 Waste Transferred 385.15 g

Empty Bottle with wash water 361.97 Washed Bottle Weight 315.44 solids left not all solids Water added 46.53 g 17.75 g 64.25

second wash 331.78 after wash/transfer 314.03 all solids transfer second time

AN-102/C-104 FEED#2 Thin layer covering bottom of bottle

Full Bottle weight 742.52 Empty Bottle Weight 361.45 Waste Transferred 381.07 grams

Empty Bottle with wash water 376.01 Washed Bottle Weight 360.11 Water added 15.90 g

AN-102/C-104 FEED#3 thin layer of solids on bottom Brown stain on bottle

Full Bottle weight 765.44 Empty Bottle Weight 363.98 Waste Transferred 401.46

total waste 1167.7 grams

Empty Bottle with wash water 386.63 Washed Bottle Weight 362.64 Water added 23.99 g

✓ 3:45 AM Stirrer turned on

Mix the total volume of waste in the large beaker. Use a volumetric flask (ball flask) to determine the density of the waste. Record the density of the AN-102/C-104 feed.

total waste 1271.85 gram total of 1041.17 gram washwater

Record Cell Temperature 31

#2

tare flask 8.930 g, flask + waste 21.384 g, waste mass 12.45 g, flask volume 10 mL

density 1.245 g/mL

Repeat in clean volumetric.

#1

tare flask 9.258 g, flask + waste 21.744 g, waste mass 1.249 g, flask volume 10 mL

density 1.249 g/mL

Average density 1.247 g/mL

Pour the waste out of the volumetric flasks back into the large beaker.

Removed 5 mL of sample from the large beaker, and filter with a disposable syringe/filter into vial # LS-01. Record the weight of sample taken.

Vial LS-01 16.830 Vial plus sample weight 23.398 sample removed 6.568 grams

While mixing add the 21 mL of 1M Sr(NO3)2 over a period of approximately 5 minutes. Record the time addition was started and completed. After the addition is complete, mix for approximately ~~30~~ minutes and remove a 5 mL sample of the slurry, noting the time the sample was removed. Filter the sample immediately with a disposable syringe/filter into vial # LS-02. Record the weight of sample taken.

DTB 10, to total 15 min

Weight 1M Sr(NO3)2 initial 121.303 Weight after addition complete 99.231 gram 22.072 g added

Addition Start Time 5:04 AM Addition Complete 5:10 Mixing Complete 5:20

Sample Removed 5:22

Vial LS-02 tare 17.003 Vial plus sample weight 23.725 sample removed 6.722 grams

With continued mixing, add the 21 mL of 1M NaMnO4 over a period of 5 minutes. Record the time addition was started and completed. After the addition is complete, mix for approximately 30 minutes and remove a 5 mL sample of the slurry, noting the time the sample was removed. Filter the sample immediately with a disposable syringe/filter into vial # LS-03. Record the weight of sample taken.

Weight 1M NaMnO4 initial 121.871 gram Weight after addition complete 99.394 grams

Addition Start Time 5:28 Addition Complete 5:36 Mixing Complete 5:44 22.477 g. added

Sample Removed 5:46 "harder to filter"

Vial LS-03 tare 16.798 Vial plus sample weight 23.320 grams sample removed 6.522 grams

One hour after the mixing the permanganate was completed, remove a 5 mL sample of the slurry, noting the time the sample was removed. Filter the sample immediately with a disposable syringe/filter into vial # LS-04. Record the weight of sample taken.

Sample Removed 6:33 "filtered OK"

Vial LS-04 tare 16.870 Vial plus sample weight 23.368 sample removed 6.498 grams

Two hour after the mixing the permanganate was completed, remove a 5 mL sample of the slurry, noting the time the sample was removed. Filter the sample immediately with a disposable syringe/filter into vial # LS-05. Record the weight of sample taken.

Sample Removed ~~7:28~~ filter/syringe plugged resampled 7:33

Vial LS-05 tare 16.921 Vial plus sample weight 25.518 g sample removed 8.597 grams

Four hour after the mixing the permanganate was completed (end of the 4 hour digest), remove a 5 mL sample of the slurry, noting the time the sample was removed. Filter the sample immediately with a disposable syringe/filter into vial # LS-06. Record the weight of sample taken.

Sample Removed 9:38 target time = 9:36

Vial LS-06 tare 16.714 g Vial plus sample weight 23.077 grams sample removed 6.363 grams

The sample is ready to be transferred to the CUF for filtration tests.

5.3 Preparation for Crossflow Filtration Tests

The CUF test matrix is shown below in Table 1. This is a modification of the test matrix used for previous RPP tests at both Battelle and SRTC. Four additional test conditions were added at higher and lower crossflow velocity and transmembrane pressure. In addition because of the strong time dependence on flux rate, the first condition will be run for three hours before proceeding to the next test conditions, and repeated at the middle and end of the matrix.

As part of the CUF installation and shakedown testing, the clean water flux was determined for the CUF at pressures of 10, 20 and 30 psi. These tests will not be conducted again, but the waste will be transferred into the CUF and testing start at test condition 1.

Follow the Operating Procedure for the CUF (TPR-RPP-WTP-058). Make sure that "0.0 Prestart" has been completed.

Proceed to "1.0 Start-Up" and check that valves are in the correct position before pouring waste into the resorvoir. Record the weight of the beaker and waste. Swirl/mix the waste slurry thoroughly before transferring to the CUF slurry reservior. There will be some solids left in the flask but try to minimize these by swirling the flask during the transfer of the final small volumes of wastes. If excessive solids remain, consult with the cognizant scientist/engineer or task leader on recovering these solids. A small amount of supernate could be pipetted from the slurry reservoir to wash these solids out of the flask. Record the method of recovery on this test instruction or in the LRB.

770.14 - 768.04 = 2.10 grams left in beaker

Record the weights of waste and beaker.

Vessel and treated waste 1963.80 g
Empty vessel 770.14 g   Weight transferred to CUF 1193.66 g

Record the level in the slurry reservoir sight glass if visible.

Height 4 3/4" inches ~ 950 ml

1167.68 grams waste
104.17 gram wash sol.
22.0772 g $Sr(NO_3)_2$
22.4779 $NaMnO_4$
1316.40 grams - 41.27 g samples
= 1275.13 g
lost 79.37 g
81.47 g to evaporation
2.1 g left in beaker

5.4 CUF Operation: Sr/TRU Precipitate Removal Test

5.4.1 Turn on the heat exchanger following "2.0 Operation of Heat Exchanger." Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in Operating Procedure using the conditions below in Table 1. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test scientist/engineer should initial and date the table below. Back pulse between test conditions, "6.0 Back Pulsing." If no filtrate flow occurs or the filtrate flow is less than 10 mL/min, discontinue testing, back pulse, and move to the next condition.

Collect a filtrate sample at 30 minutes into the testing for the conditions specified using the filtration collect sample valve, "9.0 Filtrate Sampliing." Record the weight of filtrate collected.

Table 1. Test Matrix for CUF Filtration Tests   Note time CUF Started : 10:42

	Condition	Velocity (ft/sec)	Flowrate (gpm)	TMP (psig)	Sample ID #	Sample Weight gross wt (g)	Initial/Date Completion	
Shift 1	✓1	11	3.8	40	LS-07	23.7	5-7-01	
	✓2	11	3.8	40	LS-08	25.4	5-7-01	
	✓3	11	3.8	40	LS-09	25.0	5-7-01	
	✓4	9	3.1	30	None	None	5-7-01	couldn't hit
	✓5	13	4.5	30	None	None	5-7-01	
Shift 2	✓6	13	4.5	50	LS-10 T=50 minutes	25.4	5-7-01	
	X7	9	3.1	50	~~LS-10~~	—	Step was eliminated per JG HG telecon	
	✓8	11	3.8	40	None	None	5-7-01	
	✓9	7	2.4	40	None	None	5-7-01 2037	couldn't hit
	10	15	5.2	40	None	None	5-7-01	couldn't hit
	11	11	3.8	20	None	None	5-7-01	
	12	11	3.8	60	None	None	5-8-01	
	13	11	3.8	40	LS-11	23.7 g	5-8-01	
	Dewater	TBD	TBD	TBD	~~LS-11~~		volume too low to dewater & still back pulse filter before draining	

John Geeting's home phone number was obliterated

John Geeting for telecon [redacted]

5.4.2 Conduct dewatering in the CUF, "4.0 Operation during Ultrafilter Dewatering Mode." Conduct dewatering using the preferred CUF conditions, identified by examining the data from the test matrix. Collect the filtrate in the tared bottle labeled "AN-102/C-104 CUF Filtrate." Back pulse if necessary to keep filtrate flux to acceptable levels, but the starting volume is so small that it should take less than 30 minutes to reduced the volume to the CUF limiting capacity of ~750 mL. Additional dewatering will be done using deadend filtration. RTH Volume too low to dewater in CUF

Tare weight of AN-107/C-104 CUF Filtrate Bottle ~~NOT DONE~~

Bottle and filtrate ~~1190.27 grams~~ RTH Weight of CUF Filtrate ~~Not Done~~ RTH

5.4.3 Turn off the pump and proceed to draining procedure.

5.5 Draining the Dewatered Slurry from the CUF RTH Not done - too little volume

5.5.1 Tare the 1 liter bottle labeled, "CUF Dewatered Slurry."

Weight of bottle and lid ~~188.76~~ g + hose clamps

5.5.2 Conduct the "12.0 Draining the system" operation. Collect slurry in 1 liter bottle. Make sure as much materials as possible has been collected. This material will be further dewatered using a deadend filter unit. Weigh bottle after all slurry has been removed from the CUF.

tare bottle 117.14 g

Weight of slurry, bottle and lid 1190.27 g
Weight of material collected 1073.13 g

If the CUF appears to be holding up excessive slurry, try increasing the recovery by the addition of two 50-mL volume of stabilized water (0.01M NaOH). Record weight of water added, and weight of any additional material recovered.

NOTE: Proceed with rinsing the CUF and conduct 5.7 (LRH) Dewatering using a Deadend Filtration Unit when time allows. The deadend filtration can start as soon as the initial rinse of the CUF is completed.

5.5.3 Conduct the "10.0 Rinsing the system" operation. The first rinse should be done with 1 liter of stabilized water. This liquid should be collected and ~~saved~~ in the container labeled "AN-102/C-104 CUF First Rinse." The second rinse should be done with 2 liters of filtered, stabilized water, and the final rinse with 1 liter filtered, stabilized water. The second and third rinses should be collected separately from the first in the alkaline rinse storage container. Not saved - Very little waste remained in CUF. RTH

NOTE: Once the CUF has been rinsed you may proceed to Section 5.7 (LRH) and come back and complete the CUF cleanup later.

Shift change - RTH → JGHG

5.5.4 Perform "1.0 Start-Up" operations with 1.0 liter of filtered, stabilized water.

5.5.5 Perform "6.0 Back pulsing" operations.

5.5.6 Determine the clean water flux at 20 psid transmembrane pressure and crossflow velocity of 11 ft/sec (flow rate of 3.8 GPM) following "3.0 Operation during Ultrafilter Recycle Mode" operations. Filtrate flow rate should be monitored and data collected in the operating procedure. Data should be collected for at least 20 minutes and the system should be back pulsed.

5.5.7 Compare final water flux data to initial clean water flux. The cognizant scientist/engineer will determine if Cleaning system with In-line filter (11.0) should be conducted. If in-line cleaning is performed, repeat step 5.5.6 to determine the clean water flux. Compare results to initial clean water data and determine if additional cleaning is needed. Shut off the system and conduct the "10.0 Draining the system" operation as needed.

5.5.8 Conduct the acid wash of the CUF unit with 1M HNO_3/0.1-0.2M Citric Acid as described in the CUF operating procedure (10.0) if needed to regain clean water flux. Consult the Task Manager if acid cleaning is necessary. When drained, the acidic solution should be placed in a separate container.

5.5.9 The CUF should be drained according to "12.0 Draining the system" operation and rinsed at least 3 times with filtered, distilled water to bring the pH back up to neutral. The acidic solutions should be placed in a separate container from the alkaline ones.

5.5.10 Perform "1.0 Start-Up" operations with 1.0 liter of filtered, distilled water.

5.5.11 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations using the conditions below in Table 2. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back pulsed. After each condition, the test engineer should initial and date the table below.

Table 2. Conditions for Clean Water Flux Measurement.

Condition	Velocity (ft/sec)	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	11	3.8	10	[initials] 5-8-01
2	11	3.8	20	[initials] 5-8-01
3	11	3.8	30	[initials] 5-8-01

5.5.12 Shut off the system and conduct "12.0 Draining the system," "13.0 Shutting Down," and "14.0 Lay Up." not done. completed 5-9-01

5.6 Dewatering with the Deadend Filter Unit

deadend filtration OK at first but slowed way down. 100 mL added to filter unit at 6:45A 5-8-01

5.6.1 Tare the 1-L receiver bottle/lid and filter assembly/lid of the filtration unit.

Tare of receiver bottle and lid 188.76 and hose clamps
Tare of the filter assembly and lid See log book

5.6.2 Assemble 1-L filtration unit and filter all of slurry in bottle labeled "CUF Dewatered Slurry." Solids should have settled on setting. Decant most of the supernate from the bottle and filter. The supernate should filter quite fast. When approximately half the material has been filtered, swirl the

Added ~150 mL to filter unit @ ~~0840~~ 0840 am 5-8-01 used second, then third filter unit

bottle to suspend the solids. Continue filtering the slurry. If excessive solids remain in the bottom of the bottle, small amounts of filtrate can be used to rinse the solids from the bottle. Record weight of empty bottle. Filter the solids until compacted on filter and no free liquid remains. Disassemble filter unit and weigh

Tare of receiver bottle/lid and filtrate 1202.44 Weight of filtrate 1013.68

Tare of the filter assembly/lid and wet solids Sum of 3 filters Weight of wet solids 44.938 g * See LRB # 13733 page 78 1700 HRS

34.348 + 2.87 + 7.720 = 44.938 g

5.6.3 The two 1-L bottles of filtrate, CUF filtrate and the deadend filtrate, ~~should be~~ combined to make a composite filtrate. Take duplicate 10-mL samples of the filtrate composite. Record weight of each filtrate and vial used.

No CUF Filtrate RJV

Vial LS-12 tare weight 16.870 Vial and sample 29.02 Sample weight 12.15 grams

Vial LS-13 tare weight 16.941 Vial and sample 29.0~~5~~7 Sample weight 12.13 grams

QW

5.7 Sr/TRU Solids Washing

Note: Additional filter units should be available because the filters may plug during washing.

5.7.1 The Sr/TRU solids are to be washed with three equal volumes of stabilized water (0.01M NaOH). Place the filter assembly containing wet solids on a tared receiver flask labeled wash solution. Estimate the volume of solids on the filter. Use 3 times the solids volume of wash solution but not more then 50 mL. Tare a 50 mL graduated cylinder. Add the volume of water. Record the weight. With the vacuum turned off, pour the wash water on to the wet solids, record weight of empty graduated cylinder. Allow the solids to rewet for an hour before turning the vacuum on. Then filter the solids until compacted on the filter and no free liquid remains.

Tare of filter unit See log book

Tare of wash ~~receiver~~ bottle and lid 97.92 g 97.92 + hose clamp

5.7.2 First Wash (Wash 1) target

RJV 30 mL weighed directly - cylinder tared on balance

Tare graduated cylinder 90.85 +30 mL of wash / weight of wash 28.96 g.

5.7.3 Repeat 5.7.2 for second wash and record weights. (Wash 2)

Tare graduated cylinder / RJV +30 mL of wash / RJV weight of wash 31.19 g

5.7.4 Repeat 5.7.2 for third wash and record weights. (Wash 3) RJV

Tare graduated cylinder / RJV +30 mL of wash / weight of wash 29.18 g

Tare of receiver bottle/lid and wash / RJV Total Weight of 3 washes 89.33 g

Tare of the filter assembly/lid and washed solids / RJV Weight of washed solids / RJV

See log book

20 mL ~~10~~ 5/01/01 use vial # LS-14

5.7.5 Using a pipette, transfer approximately ~~10~~ mL of the wash solution to a vial. Record the weight and sample number in Data Sheet 3 for the composite wash sample. This sample will be used for chemical and radiochemical analyses. tare vial 16.810 g sample weight 21.43 grams

(w/ lid) 38.24 g

5.7.6 Tare a jar and transfer the wet solids out of ~~the filter~~ assembly. Record the weight of wet solids in the jar and the weight of the filter assembly after the solids have been removed. Transfer a small amount of solids to SAL for percent moisture determination. Dry the small sample of solids for at least 24 hours at 105 °C and record the weight of dry solids. Transfer ~~1~~ 1.005 gram of solids to a 20 mL vial for ~~chemical~~ analyses. Record vial number and weight on Data Sheet 3 or analytical data sample sheet. energetics analyses – use vial # LS-15 Tare vial (w/ lid) 16.782 g 17.67 g full

Tare Jar 129.460 Jar plus wet solids 159.14 g Weight of wet solids 29.680 grams
Jar and dry solids weight solid not dried Amount of dry solids total jar of solids not dried

The remaining wet solids should be covered with water if they need to be stored for an extended period of time. after chemical analyses have been completed + verified

5.7.7 Retain all filtrate, wash solution, and solids for future testing. Estimate if solids quantity is large enough to conduct all of the necessary energetics and gas generation testing. Consult with the RPP-WTP contractor for prioritization of the limited solids.

5.9 Experimental Clean Up and Sample Disposition. 4 oz jar w/ lid Tare 129.459 g

Clean up all of the equipment used.

Do not discard any samples without written instructions from the task manager. Duplicate samples were collected at all sampling points, but only primary samples submitted for analyses. The duplicates are to be retained until review of the analytical data is completed and notification is writing is received to dispose of these samples. The alkaline and acid CUF wash solutions should be disposed of properly.

6.0 Sample Analysis

Follow ASR instruction for sample preparation and distribution. Note that an additional sample will need to be collected for [OH] measurement by autotitration (do not add acid to these samples, PNL-ALO-101). All subsequent sample dilution to achieve instrument or ALARA conditions are to be recorded noting both volume and mass. The data for preparation of the samples for analyses shall be recorded in a table format, or on an analytical lab data sheet.

6.1 Chemical and Radiochemical Analysis

Table 3 below shows the sample analysis list. The table lists the analyses to be performed on samples generated from this test instruction. Samples will be acid digested for ICP and radiochemical analyses. TOC/TIC, anions, and hydroxide determinations are to be performed on the samples as received. All of the collected samples are to be analyzed in duplicate. The samples are to be batched and submitted to the analytical labs. An Analytical Services Request (ASR) will be completed by the cognizant scientist/task leader and submitted to the CMC.

Table 3. Samples and Their Required Analyses

Sample #	volume	type	action	analytes	time, hrs	Comments
LS-01	5	slurry		icp/Sr/alpha	0	starting solution, baseline for decontamination calculations
LS-02	5	slurry	filter immed	icp/Sr/alpha	0.5	right after Sr addition complete, before Mn added, 30 minute is stir time
LS-03	5	slurry	filter immed	icp/Sr/alpha	1	right after the Mn addition completed.
LS-04	5	slurry	filter immed	icp/Sr/alpha	2	after 1 hour of digesting ppt
LS-05	5	slurry	filter immed	icp/Sr/alpha	3	after 2 hours of digesting ppt
LS-06	5	slurry	filter immed	icp/Sr/alpha	6	at completion of digest.
LS-07	5	filtrate		icp/Sr/alpha	0.5*	30 minutes of CUF operation, mid point of first filtration condition
LS-08	5	filtrate		icp/Sr/alpha	1.5*	midpoint of second filtration condition, 1.5 hours of CUF
LS-09	5	filtrate		icp/Sr/alpha	2.5*	midpoint of third filtration condition, 2.5 hours of CUF operation
LS-10	5	filtrate		icp/Sr/alpha	6.5*	midpoint of seventh filtration condition, 6.5 hours of CUF operation
LS-11	5	filtrate		icp/Sr/alpha	13+	pull one sample during CUF dewatering
LS-12	10	filtrate comp		all		combined CUF and deadend filtrate
LS-13	10	filtrate comp		all		combined CUF and deadend filtrate
LS-14	10	Wash		all		Combined solids wash solution
LS-15	5 g	solids		all		Washed solids, to dry and fuse for analyses

(a) Descriptions of analyses are contained in Table 4.

Table 4. Description of Analyses

Constituent	Analysis Method	PNNL Procedure No.
Acid digestion for Am-241, Sr-90 and ICP		PNL-ALO-101
Strontium-90	Separations and Beta Counting	PNL-CMC-476/408
Americium-241 (Cm)	Separation, plating and AEA	PNL-CMC-417, 496 422
Hydroxide	EPA SW-846 Modified Method, 310(3)	PNL-ALO-228
Na and total Sr (report all ICP metal listed in Table 5)	ICP-AES	PNL-ALO-211/280

Table 5. Minimum Reportable Quantities for Liquid Samples (supernatant/filtrate).

Analyte	Minimum Reportable Quantity (μg/mL)

Analyte	Minimum Reportable Quantity (μg/mL)
Al	7.5E+01
Ba	2.3E+00
Ca	1.5E+02
Cd	7.5E+00
Cr	1.5E+01
Cs	1.5E+00
Fe	1.5E+02
K	7.5E+01
La	3.5E+01
Mg	3.0E+02
Na	7.5 E+01
Ni	3.0E+01
P	6.0E+02
Pb	3.0E+02
Sr	8.7E+01
^{241}Am	7.2E-04 μCi/mL
^{90}Sr	1.5E-01 μCi/mL
Total Alpha	AEA counting
Anions	IC
TIC/TOC	
OH	0.05M

GEA

"water leach"

7.0 Important Information

Estimated density of AN-102/C-104 feed = estimated 1.26 g/mL

8.0 References

Reynolds, Dan. 2001. River Protection Project – Waste Treatment Plant Test Specification for Sr/TRU Removal and Ultrafiltration of AN-102/C-104 Waste Blend, TSP-W375-01-00003, January 31, 2001, CH2M Hill Hanford Group, Inc.

9.0 Attachments

Calculations
Reagent Prep Sheet

Calculations

Waste volume after three 25 mL bottle wash and 5 mL removed 1000 mL
Target concentration for final treated waste is 0.02M in both Sr and Mn
Use 1M solutions of both Sr(NO3)2 and NaMnO4
20.8 mL of each reagent

final waste volume = 1041 mL of ppted waste

Test #	Velcoity ft/sec	Velocity m/sec	flow rate Liter/min	flow rate gal/min
1	11	5.20	14.33	3.79
2	11	5.20	14.33	3.79
3	11	5.20	14.33	3.79
4	9	4.25	11.73	3.10
5	13	6.14	16.94	4.47
6	13	6.14	16.94	4.47
7	9	4.25	11.73	3.10
8	11	5.20	14.33	3.79
9	7	3.31	9.12	2.41
10	15	7.09	19.54	5.16
11	11	5.20	14.33	3.79
12	11	5.20	14.33	3.79
13	11	5.20	14.33	3.79
Dewater	TBD	TBD	TBD	TBD

Data Sheet 1: Operating Data

$\frac{ml}{min} = \left(\frac{ml}{s}\right)\left(\frac{60s}{min}\right)$

Date: 05-04-01
Tank Number: N/A Inhibited WATER
Filter: Mott
Test Conditions: 10 psi, 20 psi, 30 psi @ 3.8 gpm
Operator: Don Rinehart
Test Engineer: Dave Jackson

Time	Test No.	Time	Chiller ~~Temp.~~ Set Pt @ 14 °C	Slurry Temp. °C	Slurry loop Flowrate gpm	Filter Outlet Pressure psi	Permeate Pressure psi	Filter Inlet Pressure psi	Filtrate Flowrate Volume ml	Filtrate Flowrate Time m:s	Filtrate Flowrate Flowrate ml/s	Tank Level Inches	Comments
0	1	~~1710~~ 1713	27.0	24.4	3.8	10.4	0	12.0	40ml	1:09	0.58	8.25	
10		1723	27.0	25.8	3.8	10.1	0	12	40ml	1:10	0.57	–	
20		1733	25.0	24.0	3.8	10.5	0	12	40ml	1:15	0.53	–	
0	2	1750	26.5	25.5	3.7	19.7	0	22	40ml	0:34	1.18	–	
10		1800	25.0	24.7	3.7	20.5	0	22	40ml	0:38	1.05	–	
20		1810	25.0	21.1	3.7	20.4	0	23	40 ml	0:43	0.93	–	
0	3	1820	26.0	23.8	3.9	30.4	0	32	40ml	0:27	1.48	–	
10		1830	26.0	26.7	3.9	29.7	0	32	40ml	0:27	1.48	–	
20		1840	off	25.3	3.8	29.6	0	31	44	0:31	1.42	9.5	Pump Running @ 3.8 gpm
		Backpulsed & shut system down											
						DLJ 5-4-01							

Data Sheet 1: Operating Data

Date: 5-7-01
Tank Number: AN-102 Sr/TRU Precip. Solids
Filter: 0.1 µm Mott 2 ft length 3/8" ID
Test Conditions: As indicated
Operator: Halstead.
Test Engineer: Geeting

Note: this reading ~2 min after back-pulse. JG

~950 mL waste
This reading with tank isolated - JG

	Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	~~Filter~~ Outlet Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume (mL)	Filtrate Flowrate Time (sec)	Filtrate Flowrate Flowrate	Tank Level	Comments
0	1	10:55		24.7	3.84	~~40~~ 38	0	42	40 mL	48 sec	0.83 mL/sec	4 3/4	Acting like there isn't enough
10	1	11:05		25.7	3.75	38.1	0	42	40 mL	52.6	0.76		Fluid in CUF. Tough to
20	1	11:15		28.1	3.73	37.5	0	41.5	40 ml	51.3	0.78		hit set point.
30	1	11:25		22.9	3.75	39.5	0	44	40	60.9	0.66		Took sample time 11:20 (150?)
40	1	11:35		24.3	3.78	34.7	0	39	40	68.3	0.59		
50	1	11:45		25.2	3.88	37.1	0	43	40	63.0	0.63		
60	1	11:55		26.[illegible] ~~38.0~~	3.80	~~41.0~~ 38.0	ERV	~~41~~ 42	40	66	0.61		
		1202	Back pulsed (Two pulses with 1 Fill of Back pulse tank)										
0	2	12:03		28.5	3.81	37.6	0	41	50 mL	40.0	1.25	4 1/4"	Expect ~3 1/4" based
10	2	12:13		27.8	3.84	38.0	0	42	40	51.3	0.78		on initial volume and
20	2	12:23		23.1	3.80	38.2	0	43	40	62.1	0.64		Ht of 4 3/4" Indicates
30	2	12:33		22.4	3.86	38.5	0	42	40	73.7	0.54		there is air entrainment.
40	2	12:43		27.7	3.89	36.0	0	41	40	67.2	0.59		
50	2	12:53		24.2	3.78	38.9	0	42	40	73.5	0.54		
60	2	1:03		21.6	3.75	37.5	0	41	40	91.0	0.44		
	~~2~~												
		109	Back pulsed. (Two pulses with 1 Fill of back pulse tank)										

Data Sheet 1: Operating Data

Date: 5-7-01
Tank Number: AN-102 Sr/TRU precipitate
Filter: 0.1 µm Mott 2 ft length x 3/8" ID
Test Conditions: As indicated
Operator: Halstead
Test Engineer: Geeting

	Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
			17/8°F HTX	°C	gpm	psig	psig	psig	mL	sec	mL/sec		
0	3	1:10		23.1	3.77	37.3	0	~~133~~ 41	40	38.3	1.04		
10	3	1:20		27.4	3.81	38.2	0	42	40	57.7	0.69		
20	3	1:30	5.7/7.8	23.9	3.74	37.3	0	41	40	71.1	0.56		
30	3	1:40	3.9/5.6	21.3	3.73	38.0	0	41	40	83.3	0.48		
40	3	1:50	16.1/17.2	25.3	3.70	38.2	0	42	40	79.6	0.50		
50	3	2:00	9.4/11.4	26.1	3.73	~~38.0~~ 37.3	0	41	40	79.7	0.50		
60	3	2:10	1.3/3.7	22.0	3.85	39.4	0	43	~~40~~ 20	43.0	0.46		
	2:20	Backpulsed							–				
0	4	2:22		21.3	3.2	32		33	20	25.9	0.77		
10	4	2:32	19.4/20	24.2	3.04	28.3	0	31	20	40.1	0.50		
20	4	2:42		25.7	3.25	30.6	0	32	20	37.2	0.54		
30	4	2:52	8.7/10.0	20.5	3.04	28.5	0	34	20	49.6	0.40		
40	4	3:02		22.7	3.09	28.0	0	31	20	49.9	0.40		
50	4	3:12	21.1/21.6	25.3	3.23	31.4	0	35	20	44.2			
60	4	3:22	8.1/9.5	21.5	3.07	28.1	0	31	20	55.6			
			Backpulsed (Two pulses with 1 fill of back pulse chamber)										
0	5	3:34	17.1/18.2	25.8	4.45	28.2	0	33	40	61.7			Backpulse – caused flow to
10	5	3:44	6.3/8.4	24.2	4.41	29.3	0	33	40	74.8			empty tubing. Took 2 min to fill
20	5	3:54	3.2/5.2	21.1	~~4.62~~ 4.40	27.9	0	32	20	42.8	0.47		tubing before flow was noted
30	5	4:04	13.9/15.2	24.2	4.32	29.5	0	34	20	38.4	0.52		

Data Sheet 1: Operating Data

Date: 5-7-01
Tank Number: AN-102 Sr/TRU Precipitate
Filter: 0.1µm Mott 2 ft Length x 3/8" ID
Test Conditions: As Indicated
Operator: D. Rinehart
Test Engineer: DR Jackson

	Test No.	Time (PM)	Chiller Temp. (in/out)	Slurry Temp. (°C)	Slurry loop Flowrate (gpm)	Filter Outlet Pressure (psi)	Permeate Pressure (psi)	Filter Inlet Pressure (psi)	Filtrate Flowrate Volume (ml)	Filtrate Flowrate Time (sec)	Filtrate Flowrate Flowrate (ml/s)	Tank Level	Comments
40	5	4:14	10.8/12.8	26.1	4.5	28.4	0	33	20	40.8	0.49		
50	5	4:24	1.3/3.5	21.9	4.5	29.0	0	32	20	46.4	0.43		
60	5	4:34	10.6/11.8	22.4	4.5	28.4	0	33	20	45.5	0.44		
		4:50	Backpulsed										
0	6	4:53	3.5/5.8	24.2	3.2	49.3	0	50	40	42.8	0.94		Air pressure @ maximum
10	6	5:03	0.6/2.9	24.2	3.3	49.7	0	52	20	37.3	0.54		Pump incapable of higher
20	6	5:13	13.6/15.0	26.5	3.3	50.0	0	52	20	38.9	0.51		flow @ 50psig, due to
30	6	5:23	4.7/7.0	25.0	3.3	49.3	0	52	20	45.7	0.44		air header limitations and
40	6	5:33	1.0/3.6	23.3	3.3	49.8	0	53	20	47.0	0.43		low sample volume in
50	6	5:43	-1.2/1.2	22.2	3.3	50.1	0	52	20	51.8	0.39		Tank
60	6	5:55	12.4/13.8	25.3	3.3	49.5	0	52	20	54.2	0.37		Took sample LS10
			Backpulse										@ Trun = 50 minutes for run #6
0	7												Run 7 parameters
10	7												so closely matched
20	7												what was achievable
30	7												in Run #6, that it
40	7												was determined to be
50	7												unecesary to repeat
60	7												the condition parameters
			Backpulse										for a 2nd hour.

DRJ

DRJ

Data Sheet 1: Operating Data

Date: 5-7-01
Tank Number: AN-102 Sr/TRU Precipitate
Filter: 0.1 μm Mott 2 ft Length x 3/8" ID
Test Conditions: As Indicated
Operator: Don Rinehart
Test Engineer: Dave Jackson

	Test No.	Time	Chiller Temp. (°C)	Slurry Temp. (°C)	Slurry loop Flowrate (gpm)	Filter Outlet Pressure (psig)	Permeate Pressure (psig)	Filter Inlet Pressure (psig)	Filtrate Flowrate Volume (ml)	Filtrate Flowrate Time (sec)	Filtrate Flowrate Flowrate (ml/s)	Tank Level	Comments
0	8	1809	2.2/4.5	22.7	3.8	37.3	0	41.	20	25.4	0.79		
10	8	1819	7.0/8.4	21.5	3.8	39.1	0	42	20	40.2	0.50		A lot of splashing during reading
20	8	1829	17.2/19.2	25.8	3.7	38.4	0	42	20	39.2	0.51		No splashing
30	8	1839	9.3/11.3	25.9	3.6	38.5	0	42	20	43.5	0.46		No splashing
40	8	1849	4.1/5.9	21.5	3.6	38.0	0	43	20	49.3	0.41		
50	8	1859	14.6/15.7	24.3	3.6	39.5	0	43	20	46.1	0.43		
60	8	1909	13.8/15.3	26.8	3.7	39.5	0	43	20	45.3	0.44		
		1929	Back Pulse - Twice -										Backpulsed twice to clear filter completely
0	9	1937	20.4/21.0	25.6	2.4	38.1	0	40	20	19.5	1.03		
10	9	1947	9.1/10.7	23.2	2.4	39.9	0	42	20	51.5	0.39		Splashing in glass Tube
20	9	1957	10.9/11.9	21.1	2.5	41.3	0	44	20	65.6	0.30		difficult to read
30	9	2007	12.7/18.4	23.6	2.5	38.9	0	41 ~~44~~	20	62.6	0.32		No splashing -
40	9	2017	15.9/17.0	25.9	2.6	47.8	0	~~50~~ 48	20	54.9	0.36		" "
50	9	2027	7.8/9.0	20.8	2.5	44.1	0	45	20	68.9	0.29		" "
60	9	2037	15.9/16.6	22.4	2.4	40.6	0	42	20	70.3	0.28		" "
		2054	Backpulsed Twice										2 Backpulses Req'd.
0	10	2104	-.2/2.1	22.3	3.6	39.5	0	42	20	24.7	~~24.7~~ 0.81		No Splashing
10	10	2114	10.5/12.0	24.3	3.6	39.2	0	43	20	43.8	0.46		" "
20	10	2124	13.4/15.0	27.6	3.6	39.9	0	43	20	44.8	0.45		Splashing - hard to read
30	10	2134	2.7/5.1	24.0	3.6	40.2	0	43	20	50.0	0.40		

Data Sheet 1: Operating Data

Date: 5-7-01
Tank Number: AN-102 Sr/TRU Precipitate
Filter: 0.1 μm Mott 2ft length × 3/8" ID
Test Conditions: As indicated
Operator: Don Rinehart
Test Engineer: Dave Jackson

	Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
40	10	2144	0.5/2.6	22.4	3.7	40.3	0	43	20	53.0	0.38		Splashing - Hard to read
50	10	2154	14.4/15.6	25.7	3.7	40.8	0	43	20	50.0	0.40		Hard to Read - splashing
60	10	2204	6.9/9.0	25.7	3.7	40.7	0	44	20	53.2	0.38		
		2228	Back Pulse										
0	11	2229	15.5/16.4	23.3	3.8	18.7	0	22	20	38.3	0.52		~~N~~ Splashing
10	11	2239	7.4/8.9	21.4	3.6	18.0	0	21	20	67.8	0.30		No Splash
20	11	2249	10.3/11.3	20.3	3.7	19.6	0	23	20	62.6	0.32		No Splash
30	11	2259	17.2/18.0	23.7	3.9	20.5	0	24	20	58.3	0.34		" "
40	11	2309	7.5/9.2	22.6	3.9	20.1	0	24	20	67.3	0.30		" "
50	11	2319	6.2/7.5	18.8	3.7	19.1	0	24	20	76.2	0.26		
60	11	2329	15.2/16.1	22.2	3.8	19.5	0	23	20	70.8	0.26		
		2340	Back Pulse										
0	12	2342	7.3/9.2	24.8	2.8	60.2	0	62	20	21.8	0.92		No Splash
10	12	2352	0.4/2.8	23.9	2.8	61.3	0	64	20	52.7	0.38		
20	12	2402	-2.9/-0.3	22.5	2.8	63.0	0	65	20	58.6	0.34		
30	12	2412	4.4/6.2	22.9	2.8	60.8	0	64	20	64.3			
40	12	2422	13.4/14.8	27.2	2.8	61.0	0	64	20	61.0			
50	12	2432	4.4/6.5	25.3	2.8	60.0	0	64	20	64.9			
60	12	2442	-0.2/2.1	23.5	2.8	61.2	0	64	20	71.0			
			Back Pulse										entrained air when back pulse chamber filled caused both pressure & velocity to drop during chamber filling

RTH 5-8-01

Data Sheet 1: CUF Operating Data

Date: 5-7-01
Tank Number: AN-102 Sr/TRU Precipitate
Test Conditions: As Indicated
Operator: ~~Don Rinehart~~ Tim Reining
Test Engineer: ~~Dave Jackson~~ Rich Hallen

14.5/6.8 DJV

Note 2503 = 1:03 on 5-8-01

" NOTE "
SAMPLE AT ~ 30 minutes

	Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	~~Permeate~~ Filter DJV Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Time	Flowrate	Tank Level	Comments
0	13	25:03	~~25~~ 14.5	24.5	3.85	38.6	42	20	26.8			
10	13	2513	18.3/19.4	28.0	3.86	36.9	40	20	43.5			
20	13	2523	3.6/5.9	24.5	3.80	37.0	42	20	52.2			
30	13	2533	10.4/11.7	23.5	3.87	38.2	43	20	56.8			take sample
40	13	2543	17.7/18.8	27.1	3.74	37.1	41	20	53.9			
50	13	25:53	6.2/8.3	25.5	3.79	37.2	42	20	~~60~~ 61.0			
60	13	2603	7.5/9.1	23.2	3.75	37.9	42	20	63.0			
	Backpulse before drain											

Data Sheet 1: CUF Operating Data

Date: 5-08-01
Tank Number:
Test Conditions: Clean water flux (0.01M NaOH)
Operator: Halstead
Test Engineer: Geeting

	Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate (gpm)	Permeate Pressure	Filter Inlet Pressure (psig out/inlet)	Filtrate Flowrate Volume (mL)	Filtrate Flowrate Time (sec)	Filtrate Flowrate Flowrate	Tank Level (Inches)	Comments
0	Clean water	8:38		29.9	3.71	0	11.6/13	40mL	20.25sec	1.98	4 3/4	
10		8:48		23.3	3.78	0	10.1/12	40	32.5sec	1.23		
20		8:58		24.2	3.81	0	10.0/12	40	33.0	1.21		
		9:02	Backpulse	~~26~~	~~3.82~~	~~0~~	~~20.2/~~	~~40~~	~~14~~ 14.0			
0		9:03		26.0	3.82	0	20.2/22	40	14.0	2.86		
10		9:13		27.2	3.80		21/23	40	17.0	2.35		
20		9:23		24.7	3.77	0	20.8/23	40	19.0			
		9:27	Backpulse	~~25~~			~~29.6~~					
0		9:28		25.8	3.82	0	29.6/32	40	10.4	3.84		
10		9:38		27.4	3.86		29.7/32	40	12.8	3.12		
20		9:48		24.4	3.84		29.2/32	40	14.4	2.77		

5-7-01

Data Sheet 3: Sample Log

Tank Number 4N-102 Sr/TRU precip. Solids.

Filter 0.1 μm mott

Test Conditions

when taking sample alignment off. ~3 mL

Condition #

Sample Number	Date	Time	Sample Type	Test Instruction Step	Tare Weight (g)	Total Mass (g)	Mass of Sample (g)	Comments	Operators Initials
LS-07	5-7-01	11:20	permeate	1		23.7		Permeate Spilled	[illegible]
LS-08	5-7-01	12:40	permeate	2		25.4			
LS-09	5-7-01	1:42	permeate	3		25.0		Took sample on step 6 @ 50 minutes	② [illegible]
LS-10	5-7-01	5:50	permeate	6		25.4			
LS-11	5-8-01	25:39	permeate	13		23.69			

Data Sheet 3: Sample Log

tare sheet
- outside hot cell
Balance # 380-06-01
SN N44766 2/2002 013

Tank Number AN-102/C-104 Blended Waste

Test Number Sr/TRU Removal & Filtration TI-073

Sample Number	Date	Time	Sample Type	Tare Weight (g)	Total Mass (g)	Mass of Sample (g)	Operators Initials
LS-01				16.8299			
LS-02				17.0034			
LS-03				16.7977			
LS-04				16.8699			
LS-05				16.9210			
LS-06				16.7139			
LS-07				16.7194			
LS-08				16.9108			
LS-09				16.9593			
LS-10				16.7980			
LS-11				16.7863			
LS-12				16.8699			
LS-13				16.9412			
LS-14				16.8115			
LS-15				16.7834			
LS-16				16.7970			
1M $Sr(NO_3)_2$				98.8163			
1M $NaMnO_4$				98.9733			
10mL vol. 1				9.2571			
10mL vol. 2				8.9298			
250mL Wash composite bottle				~~39.550~~			
		w/o	Lid	39.4670			
		Bottle	Lid	11.2373			
complete filter top + cap				39.2382			

Treatment of AN-102/C-104 Blended waste at target 0.02M concentration
per liter of waste

1M Sr	0.021 Liter or	0.020 moles
1M MnO4	0.021 Liter or	0.020 moles

Balance number: N44766.
Calibration date: 2/2002
due

Makeup Sr(NO3)2 solution 25 mL
use Sr(NO3)2
211.63 grams/mole (FW) (lot # 984987)
Tare 25 mL vol. flask 21.6429 grams
add 5.29075 grams Sr(NO3)2 to volumetric + 5.2923 grams
actual weight of Sr(NO3)2 added 5.2923 grams
fill to mark on volumetric + H2O 28.8230 grams
calculate density 1.153 g/mL
Tare bottle 98.8164 grams
Label bottle with ID, add 21 mL of 1 M solution to bottle 24.2211 grams
target 24.21g
total weight 123.0376g

Makeup NaMnO4 solution 25 mL
use NaMnO4.H2O
159.94 grams/mole (FW) (lot #A010675901)
Tare 25 mL vol. flask 21.9509 grams
add 3.9985 grams NaMnO4 to volumetric grams
actual weight of KMnO4 added 3.9970 grams
fill to mark on volumetric + H2O 27.2573 grams
calculate density 1.090 g/mL
Tare bottle 98.9733 grams
Label bottle with ID, add 21 mL of 1 M solution to bottle 22.9740 grams
target 22.90 grams

Date prepared: R. Hallen
Prepared by: 5/04/01
Work Package Number: W57984

total weight 121.9474

Title Test Instruction for In-Cell Shake down Testing of CUF.
Test Instruction: TI-RPP-WTP-077 Rev. 0
Date: 5/02/01.
Supporting Document: TRP-RPP-WTP-058, PNNL Operating Procedure for Ultrafiltration Testing.

Approved:

Author [signature] 5-2-01 Technical Reviewer [signature] 05-02-01

Materials:

- One sample vial
- 2 liters of 0.01 M NaOH
- Pipettes
- ~20 mL of bleach (5% sodium hypochlorite)
- Tank level vs. volume data
- Rotometer calibration data
- Stop watch

Instructions: The test engineer has the authority to make modifications to this test instruction. Any deviations from the test plan should be noted and initialed.

1. Verify all fittings on the CUF are tight.

   Verified complete [signature] 5-3-01 Any Issues: 1) Did not use a wrench to check fittings due to difficulty in reaching them. physically "shook" each joint to check it. All appeared okay.

2. Follow Prestart Instructions, Section 0.0.

   Verified complete [signature] 5-3-01 Any Issues: 1) Step 8 - We need details on how to label data files & locate them. File name is Getting1.WBW - verify

3. Verify that the computer, flowmeter, TC 1-4, RTD, and the PT are operating correctly.

   Verified complete [signature] 5-3-01 Any Issues: 1) Need to verify polarity on T3

4. Follow Start-up Instructions 1.0. Use 2 liters of 0.01 M NaOH in step 1.6. Record mass or volume of simulant added. 2 Liters

   Verified complete [signature] 5-3-01 Any Issues: 1) Need to add V29 & V30 to TPR-RPP-WTP-058 These are additional isolation valves on air supply line 2) Need to put a bottle in for Slurry Mix Tank drain

5. Inspect all fittings for leaks (including the chiller lines). Note any that are not corrected.

   Verified complete [signature] 5-3-01 Any Issues: Found 3 leaks - corrected each & shutdown system. Lost about 1400 ml

6. Operate level indicator. Level = 8.875. Compare level with expected level. When complete turn level indicator off. (Added 2 Liters, .01 M NaOH

   Verified complete [signature] 5-3-01 Any Issues: None

7. Turn on agitator. Verify that it can be controlled with variac.

   Verified complete [signature] 5-3-01 Any Issues: None

① ⓠ 8-4-01

8. Operate system in recycle mode IAW section 3.0. Run each condition for ~~30~~ ~~10~~ 20 minutes. Back pulse before each run. Targeted conditions are indicated in the table below. Check for leaks. Record data on Data sheet 1. (Note: Flux rate during cold testing at 20 psig was 186 mL/min [3.1 mL/sec] after 20 minutes of running.)

5-03-01

Condition	Flowrate (gpm)	TMP (psig) 10 min	Tmp 20 min step 8b	Initial/Date Completion	TEMP.
1 1325	3.8	10	10	5-03-01	31.9 °C
2	3.8	.17	20	5-03-01	~~33.8~~ ~~36.7 °C~~
3	3.8	25	30	5-03-01	33.8 37.8 °C

32.7-33

5-4-01

9. Run system in recycle mode IAW section 3.0. Run each condition for 10 minutes. Backpulsing is not required.

Condition	Flowrate (gpm)	TMP (psig)	Initial/Date Completion
1	4.5	50	5-4-01
2	5.2	40	5-4-01
3	3.8	60	5-4-01

* These conditions require the max. available supply of air: P = 65 psi, Air Flow = 10 SCFM

10. Take a sample IAW section 7, 8, and 9. After each sample, dump contents back into slurry reservoir.

Verified complete 5-4-01 Any Issues: • Pipette needs to be able to pull 3 ml per pipette, preferably 5 ml • Longer pipette needed to reach the 2 L level use SAMCO Transfer Pipettes Cat. 263 Approx 5-6 ml/cap

HOLD 5-4-01 per JGHG

~~11. Operate the in-line filter IAW section 11.~~ Delete

Verified complete delete Any Issues:

12. Turn off Chiller IAW section 2.0. Run system at 3.8 gpm and 30 psig. Turn on heat tape. Put in a setpoint of 40 C on the controller and let it heat up. Note time to heat up. Start 1635 Stop 1650. After 40 C has been reached turn on chiller IAW section 2.0. Set the chiller set point to 25 C. Note time to cool back down to 25. Start 1650. Stop 1703. Control temperature to 25 C. Note deviations from 25 C.

Verified complete 5-4-01 Any Issues: Set Chiller SP to 25 °C @ 3.8 gpm + 30 psig.

13. Shut system down in accordance with Section 13.

Verified complete 5-4-01 Any Issues: NONE

N/A 14. If AN-102 testing is still scheduled within 1 week skip this step and proceed to step 14; otherwise lay up system IAW section 14.

5-4-01

Verified complete N/A Any Issues: N/A

15. Copy computer file onto disk and verify that it was saving data properly.

Verified complete 5-4-01 Any Issues: John took disk to check data

16. Drain System per Section 12.0

Complete 5-4-01

Appendix B

Analytical Data Reports – Chemical and Radiochemical Analyses

Battelle
. . . Putting Technology To Work

Project No. 42040

Internal Distribution
File/LB

Date November 14, 2002

To R. Hallen

From L. R. Greenwood LRG

Subject Radiochemical Analyses for AN-102/C-104 Blend – ASR 6107

Samples of the filtrates from tanks AN-102/C-104 blend were analyzed for gamma emitters, ^{90}Sr, ^{99}Tc, alpha/AEA, U, and Am/Cm according to ASR 6107. The samples were acid digested or fused in the hot cells according to procedures PNL-ALO-128 or –115 and aliquots were delivered to the laboratory for analysis. The acid digestions were performed in four different batches in the hot cells, each batch having a separate process blank. The one solid sample, LS-16, was prepared in the hot cells by KOH-KNO_3 fusion. The attached reports list measured analyte activities in the original sample material in units of uCi/g. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. Laboratory and process blank values given with each analysis are the best indicators of the method detection limits, taking into account the actual sample sizes and counting times used for each analysis.

Gamma Spectrometry

Sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450. The samples were prepared in the hot cells in two batches, each having its own process blank, as indicated by the batch numbers on the data table. Since no sample preparation was involved in the counting laboratory, no additional blanks or spikes were prepared for these analyses other than the standard laboratory control samples and background counts. In order to meet the requested detection limits, diluted aliquots of the hot cell preparations were counted for periods of 3 to 14 hours. All of the samples showed the presence of significant ^{137}Cs activity. Some of the samples also showed the presence of ^{60}Co, ^{154}Eu, ^{155}Eu, and ^{241}Am. The MRQ values for extended counting time GEA were met in all cases, and detection limits are listed in the tables. The hot cell process blanks showed the presence of ^{137}Cs; however, the activities were well below the 5% criteria specified in the QA plan. Sample duplicates showed good repeatability with the exception of sample LS-16 with a RPD value of 37% for ^{60}Co and a mean difference (MD) of 2.23, well outside of the required agreement of 20% for the RPD or 1.96 for the statistical MD test that takes into account the uncertainties on the measurements. The reason for the large disagreement for this one isotope (^{137}Cs and ^{154}Eu results are in acceptable agreement) is not known, but may suggest ^{60}Co contamination or heterogeneity in the sampling in the hot cells. The activities measured for ^{241}Am are in good agreement with the Am/AEA results reported below for sample LS-16.

Strontium-90

The Sr separation was performed according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr. The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). The samples were prepared in the hot cells in five batches and analyzed in two batches in the laboratory, as shown by the batch numbers on the report for QA assessment. Some of the separated Sr fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples in addition to the beta correction from ^{85}Sr. In most cases, the ^{137}Cs correction was small (<1%) compared to the activity in the samples, except for sample LS-14 where the correction was 9%. The laboratory blank BLK-1006 for the first batch showed ^{90}Sr activity up to 15% of the activity in the samples, higher than the QA criteria of 5% of the sample activity. However, the uncertainty on the ^{90}Sr activity in this lab blank is very high (33%) due to the very large ^{137}Cs and ^{85}Sr beta corrections for this sample. Consequently, this lab blank for the first batch of samples is not useable for assessing laboratory contamination and the hot cell process blanks should be used instead. The blank BLK-1010 for the second laboratory batch as well as the five hot cell process blanks did not show any significant (below the QC criteria of5%) ^{90}Sr contamination. Sample duplicates showed good repeatability in all cases except for sample LS-16 where the RPD value slightly exceeded the QC value of 20% and the MD value exceeded the statistical level of 1.96., We note that the duplicates for this sample also fail the RPD and MD tests for ^{99}Tc and ^{60}Co, suggesting heterogeneity of these duplicates. The blank spike and matrix spike yields ranged from 97% to 104%. All of the samples showed the presence of ^{90}Sr at levels that were significantly less than the requested MRQ values.

Total Uranium

Total uranium was measured in samples LS-12 to LS-14 and LS-16 according to procedure PNNL-ALO-4014 using Kinetic Phosphoresence Analysis (KPA). The samples were prepared in two batches in the hot cells, as indicated by the batch number on the data report. Uranium was detected in both the hot cell preparation blanks; however, the levels were well below the 5% criteria for samples in each batch as specified in our QA plan. Sample duplicates and a lab replicate showed good repeatability. Since the analyses were performed on the samples as received from the hot cells, no sample spikes were required. LCS samples gave uranium results at 99% and 102% of the expected values. All of the measured uranium values were well below the requested MRQ values.

Total Alpha with Alpha Energy Analysis

The samples were prepared in the hot cells in five separate batches and analyzed in the laboratory in one batch for both total alpha activity and total alpha energy analysis. The total alpha activity was determined by direct plating small aliquots of the hot cell acid-digested or fused samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum ZnS scintillation detectors according to RPG-CMC-408. Alpha energy analyses were performed on all samples according to procedure RPG-CMC-422. Peaks were observed due to $^{234}U+^{237}Np$, $^{239}Pu+^{240}Pu$, $^{238}Pu+^{241}Am$, $^{243}Cm+^{244}Cm$, and ^{242}Cm. The sums of the individual alpha emitters are generally in fair agreement with the total alpha data indicating minimal losses due to alpha self-absorption. The RPD values exceed the QC criteria of 20% for duplicates of samples LS-01

(^{242}Cm), LS-02 (^{242}Cm), LS-04 (total alpha), LS-05 ($^{243+244}Cm$), LS-06 (total alpha), LS-07 (^{242}Cm), LS-08 (total alpha), LS-09 (^{242}Cm), LS-10 (total alpha), and LS-11 (242Cm).. In almost all of these cases, the data pass the mean difference statistical test (MD < 1.96 at the 95% confidence level) in our QA plan indicating that the results are acceptable when the uncertainties are taken into account. The sole exception is 243+244Cm for sample LS-05 where the MD value is 3.15. The reason for this difference is not known. The sum of the alpha emitters is deemed to be more reliable than the total alpha valuessince the total alpha measurements have higher statistical uncertainties and may suffer from alpha absorption effects. The LCS and matrix spike recoveries were 102% and 105%, respectively.

Most of the hot cell preparation blanks and the laboratory blank did not show any significant alpha contamination. However, for the hot cell acid digestion batch containing samples LS-01, LS-12, LS-13, and LS-14, the process blank (labeled 01-1014PB) contained high alpha contamination relative to the samples. The blank activity is acceptable at only 3% of the alpha activity in sample LS-01, but it is not acceptable (> 5%) compared to the other samples. The alpha activity in the hot cell process blank is 16% of that in sample LS-12, 23% of LS-13, and 115% of sample LS-14, such that these data would not normally be acceptable. However, it should be pointed out that these sample activities are well below the requested MRQ value of 0.23 uCi/ml, such that the data might be acceptable to the project in spite of the additional uncertainty due to the high levels of alpha contamination. The total alpha activity for this hot cell blank (01-1014PB) is only 30% of the sum of the individual alpha emitters detected by AEA and the RPD value is 88%. Although the reason for this disagreement is unknown, the alpha/AEA result is more reliable than the total alpha result since the alpha/AEA sample was re-prepped from a fresh sample aliquot, and its counting data agreed with the original alpha/AEA data.

Americium and Curium

The Am/Cm separations were performed for samples LS-12 to LS-14 and LS-16 according to PNL-ALO-417. The separated fractions were precipitation plated according to PNL-ALO-496 and the samples were counted by alpha spectrometry according to PNL-ALO-422. The samples were prepared in the hot cells in two separate batches and analyzed in the laboratory in one batch. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. As discussed above for the total alpha results, the hot cell process blank (01-1014 PB) that was prepared with samples LS-12 to LS-14 was contaminated with alpha activity at unacceptable levels (>5%). There was no significant contamination of the hot cell process blank prepared with the solids sample LS-16. The Am/Cm AEA results are generally in good agreement with the total alpha AEA results indicating that there is little ^{238}Pu in these samples. The only exception is for the contaminated hot cell process blank (01-1014 PB), which clearly has a different isotopic mix than the samples. For example, the ratio of $^{238}Pu+^{241}Am$ to that of $^{239}Pu+^{240}Pu$ in the process blank is lower than seen in the samples whereas the $^{243+244}Cm$ content in the process blank is much higher than seen in the samples. Such isotopic differences are also indicative of hot cell contamination of the samples. The LCS and matrix spike recoveries were 99% and 100%, respectively. RPD values were acceptable except for the ^{242}Cm RPD of 21% for sample LS-12 The counting statistics are quite high for this sample such that the MD (mean difference calculated according to our QA plan) value is less than the required value of 1.96, indicating that the measured activities are in good statistical agreement. Most of the sample activities were well below the requested MRQ values for Am and Cm.

^{99}Tc

The technetium in the solids sample LS-16 was chemically separated for analysis according to procedure PNL-ALO-432. The separated fractions were then counted according to procedure RPG-CMC-408. No activity was detected in either the hot cell process blank or the laboratory blank. The RPD value was 23%, exceeding the QC criteria of 20%, suggesting some heterogeneity in the sample duplicates. The MD statistical calculation also exceeds the required value of 1.96 indicating that the two measurements are not in agreement. As noted previously, these sample duplicates also fail the RPD and MD tests for ^{90}Sr and ^{60}Co suggesting heterogeneity in the duplicate sampling process. The matrix spike recovery of 79% was within the QC requirement of 75%, although the lower than normal recovery was probably due to the high salt content from the fusion preparation. The LCS recovery was 95%. The ^{99}Tc activities were well below the requested MRQ values.

Review:
C Soderquist

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

01-1003

11/14/02
Rev. 1

Client : R. Hallen

Cognizant Scientist: LRGreenwood   Date : 11/14/02

Concur : C Soderquist   Date : 11-25-02

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Hot Cell Batch*	Cr-51 Error %	Fe-59 Error %	Co-60 Error %	Nb-95 Error %	Ru-103 Error %	Ru-106 Error %	Sn-113 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Ce-144 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-1014PB Process Blank	1	<6.E-4	<9.E-5	<5.E-5	<5.E-5	<6.E-5	<5.E-4	<8.E-5	<2.E-4	<5.E-5	<6.E-5	6.99E-3 ± 3%	<5.E-4	<2.E-4	<2.E-4	<3.E-4	<8.E-4
01-1014 LS-12	1	<9.E-2	<3.E-3	3.15E-2 ± 2%	<2.E-3	<1.E-2	<7.E-2	<4.E-3	<4.E-2	<3.E-2	<2.E-3	1.27E+2 ± 2%	<4.E-2	<1.E-3	1.90E-2 ± 6%	<2.E-2	<2.E-2
01-1014 DUP LS-12 DUP	1	<9.E-2	<3.E-3	3.14E-2 ± 2%	<2.E-3	<1.E-2	<7.E-2	<4.E-3	<4.E-2	<3.E-2	<2.E-3	1.27E+2 ± 2%	<5.E-2	<1.E-3	1.84E-2 ± 6%	<2.E-2	<2.E-2
RPD				0%								0%			3%		
01-1015 LS-13	1	<6.E-2	<2.E-3	3.22E-2 ± 2%	<1.E-3	<8.E-3	<5.E-2	<3.E-3	<3.E-2	<2.E-2	<1.E-3	1.32E+2 ± 2%	<3.E-2	<9.E-4	2.01E-2 ± 4%	<1.E-2	<1.E-2
01-1016 LS-14	1	<2.E-2	<3.E-4	5.07E-3 ± 2%	<2.E-4	<2.E-3	<1.E-2	<4.E-4	<7.E-3	<7.E-4	<2.E-4	2.42E+1 ± 3%	<1.E-2	<3.E-4	2.25E-3 ± 7%	<6.E-3	<2.E-2
01-1017PB Process Blank	2	<2.E-2	<2.E-3	<2.E-3	<1.E-3	<2.E-3	<1.E-2	<3.E-3	<5.E-3	<2.E-3	<2.E-3	8.64E-2 ± 3%	<1.E-2	<7.E-3	<4.E-3	<8.E-3	<1.E-2
01-1017 LS-16	2	<4.E-1	<2.E-2	4.88E-2 ± 7%	<1.E-2	<5.E-2	<3.E-1	<3.E-2	<2.E-1	<6.E-2	<2.E-2	1.37E+2 ± 3%	<4.E-1	<5.E-2	4.46E+0 ± 2%	2.52E+0 ± 5%	3.82E+0 ± 5%
01-1017 DUP LS-16 DUP	2	<4.E-1	<2.E-2	7.07E-2 ± 5%	<1.E-2	<5.E-2	<3.E-1	<2.E-2	<2.E-1	<6.E-2	<1.E-2	1.48E+2 ± 3%	<3.E-1	<4.E-2	3.84E+0 ± 2%	2.23E+0 ± 5%	3.42E+0 ± 6%
RPD MD				37% 2.23								8%			15%	12%	11%

*The samples were prepared in two batches in the hot cells using an acid digestion for batch 1 (liquids) and a KOH fusion for batch 2 (solids).

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

11/14/02

Client : R. Hallen

Rev. 1

Cognizant Scientist: LR Greenwood　　Date : 11/14/02

Concur : C Soderquist　　Date : 11-25-02

Measured Activities (uCi/g) with 1-sigma error

ALO ID / Client ID	Sr-90 Error +/-	Hot Cell Batch*	Laboratory Batch**
01-1003 LS-01	2.01E+1 ± 3%	1	1
01-1003 DUP LS-01 DUP	2.03E+1 ± 3%	1	1
RPD	1%		
01-1004 PB Process Blank	5.58E-2 ± 3%	2	1
01-1004 LS-02	3.07E+0 ± 4%	2	1
01-1004 DUP LS-02 DUP	3.61E+0 ± 4%	2	1
RPD	16%		
01-1005 LS-03	1.88E+0 ± 5%	2	1
01-1005 DUP LS-03 DUP	1.93E+0 ± 5%	2	1
RPD	3%		
01-1006 LS-04	1.73E+0 ± 5%	2	1
01-1006 DUP LS-04 DUP	1.82E+0 ± 5%	2	1
RPD	5%		

ALO ID / Client ID	Sr-90 Error +/-	Hot Cell Batch*	Laboratory Batch**
01-1006 Lab DUP LS-04 DUP	1.65E+0 ± 4%	2	1
01-1007 LS-05	1.75E+0 ± 5%	2	1
01-1007 DUP LS-05 DUP	1.79E+0 ± 5%	2	1
RPD	2%		
01-1008 PB Process Blank	1.88E-2 ± 3%	3	1
01-1008 LS-06	1.68E+0 ± 5%	3	1
01-1008 DUP LS-06 DUP	1.56E+0 ± 6%	3	1
RPD	7%		
01-1009 LS-07	3.33E+0 ± 4%	3	1
01-1009 DUP LS-07 DUP	3.14E+0 ± 4%	3	1
RPD	6%		

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Sr-90 Error +/-	Hot Cell Batch*	Laboratory Batch**
01-1010 LS-08	2.92E+0 ± 4%	3	2
01-1010 DUP LS-08 DUP	3.06E+0 ± 4%	3	2
RPD	5%		
01-1011 LS-09	2.67E+0 ± 3%	3	2
01-1011 DUP LS-09 DUP	2.71E+0 ± 3%	3	2
RPD	1%		
01-1012 PB Process Blank	6.26E-2 ± 3%	4	2
01-1012 LS-10	2.18E+0 ± 3%	4	2
01-1012 DUP LS-10 DUP	2.55E+0 ± 3%	4	2
RPD	16%		
01-1012 Lab Dup LS-10 DUP	2.19E+0 ± 3%	4	2
01-1013 LS-11	1.99E+0 ± 3%	4	2
01-1013 DUP LS-11 DUP	2.04E+0 ± 3%	4	2
RPD	2%		

ALO ID Client ID	Sr-90 Error +/-	Hot Cell Batch*	Laboratory Batch**
01-1014 PB Process Blank	6.52E-2 ± 3%	1	2
01-1014 LS-12	1.28E+0 ± 3%	1	2
01-1014 Dup LS-12 DUP	1.40E+0 ± 3%	1	2
RPD	9%		
01-1015 LS-13	1.40E+0 ± 3%	1	2
01-1016 LS-14	3.26E-1 ± 5%	1	2
01-1017 PB Process Blank	1.44E-1 ± 4%	5	2
01-1017 LS-16	1.75E+3 ± 3%	5	2
01-1017 DUP LS-16 DUP	1.40E+3 ± 3%	5	2
RPD MD	22% 2.60		
MS-1006 MS-1012	104% 97%	N/A N/A	1 2
BS-1003 BS-1010	101% 103%	N/A N/A	1 2
Blk-1006	2.38E-1 ± 33%	N/A	1
Blk-1010	<9.E-2	N/A	2

*The samples were prepared in the hot cells in 5 different batches.
**The samples were analyzed in the laboratory in two separate batches

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Battelle Pacific Northwest Laboratory
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Chemical Measurements Center

11/14/02

Client : R. Hallen

Rev. 1

Cognizant Scientist: LRGreenwood Date : 11/14/02

Concur : C. Soderquist Date : 11-25-02

ALO ID Client ID	Uranium µg/g ± 1s	Hot Cell Batch*
01-1014 PB Process Blank	1.06E-1 ± 2%	1
01-1014 LS-12	2.20E+1 ± 4%	1
01-1014 Dup LS-12 DUP	2.17E+1 ± 4%	1
RPD	1%	
01-1015 LS-13	2.23E+1 ± 4%	1
01-1016 LS-14	3.15E+0 ± 2%	1
01-1016 Rep LS-14	3.20E+0 ± 2%	1
RPD	2%	
01-1017 PB Process Blank	1.82E+0 ± 2%	2
01-1017 LS-16	3.25E+2 ± 3%	2
01-1017 DUP LS-16 DUP	2.97E+2 ± 3%	2
RPD	9%	
Pre-run Post-run	102% 100%	
Blank	<2.E-5	

*The samples were prepared in two batches in the hot cells.

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Chemical Measurements Center

11/14/02

Client : R. Hallen

Rev. 1

Cognizant Scientist: LRGreenwood Date : 11/14/02

Concur : C. Soderquist Date : 11-25-02

Measured Activities (µCi/g) with 1-sigma error

ALO ID / Client ID	Total Alpha ± 1s	Alpha Energy Analysis: U-234+ Np-237 ± 1s	Pu-239+ Pu-240 ± 1s	Pu-238+ Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of alpha emitters	Hot Cell Batch
01-1003	5.90E-2	3.23E-4	2.02E-3	5.02E-2	2.43E-3	2.16E-4	5.52E-2	1
LS-01	± 6%	± 16%	± 6%	± 2%	± 6%	± 19%	± 2%	
01-1003 DUP	5.73E-2	3.58E-4	1.93E-3	4.73E-2	2.36E-3	1.70E-4	5.21E-2	1
LS-01 DUP	± 6%	± 12%	± 5%	± 2%	± 5%	± 17%	± 2%	
RPD	3%	10%	5%	6%	3%	24%	6%	
MD						0.46		
01-1004 PB	3.41E-4	7.82E-7	3.42E-5	2.05E-4	9.81E-5	<4.E-7	3.38E-4	2
Process Blank	± 7%	± 28%	± 4%	± 2%	± 2%		± 1%	
01-1004	3.57E-2	2.17E-4	1.87E-3	3.38E-2	1.93E-3	9.69E-5	3.79E-2	2
LS-02	± 7%	± 16%	± 6%	± 2%	± 5%	± 25%	± 2%	
01-1004 DUP	4.03E-2	2.47E-4	1.56E-3	3.28E-2	1.69E-3	1.47E-4	3.64E-2	2
LS-02 DUP	± 7%	± 16%	± 6%	± 2%	± 6%	± 20%	± 2%	
RPD	12%	13%	18%	3%	13%	41%	4%	
MD						0.66		
01-1005	1.16E-2	1.66E-4	7.57E-4	9.51E-3	8.04E-4	5.87E-5	1.13E-2	2
LS-03	± 13%	± 8%	± 4%	± 2%	± 4%	± 14%	± 2%	
01-1005 DUP	1.13E-2	1.75E-4	8.55E-4	9.80E-3	7.93E-4	7.11E-5	1.17E-2	2
LS-03 DUP	± 15%	± 8%	± 4%	± 2%	± 4%	± 13%	± 2%	
RPD	3%	5%	12%	3%	1%	19%	3%	
01-1006	8.92E-3	1.78E-4	7.54E-4	8.10E-3	6.54E-4	4.45E-5	9.73E-3	2
LS-04	± 16%	± 8%	± 4%	± 2%	± 4%	± 16%	± 2%	
01-1006 DUP	1.44E-2	1.49E-4	6.40E-4	8.46E-3	6.97E-4	3.73E-5	9.98E-3	2
LS-04 DUP	± 12%	± 10%	± 5%	± 2%	± 5%	± 21%	± 2%	
RPD	47%	18%	16%	4%	6%	18%	3%	
MD	1.22							

Measured Activities (µCi/g) with 1-sigma error

ALO ID Client ID	Total Alpha ± 1s	Alpha Energy Analysis U-234+ Np-237 ± 1s	Pu-239+ Pu-240 ± 1s	Pu-238+ Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of alpha emitters	Hot Cell Batch
01-1006 Lab DUP LS-04	N/A	2.15E-4 ± 9%	7.27E-4 ± 5%	8.99E-3 ± 2%	7.25E-4 ± 5%	6.83E-5 ± 16%	1.07E-2 ± 2%	2
01-1007 LS-05	1.12E-2 ± 14%	1.62E-4 ± 8%	7.64E-4 ± 4%	9.06E-3 ± 2%	1.07E-3 ± 3%	5.22E-5 ± 15%	1.11E-2 ± 2%	2
01-1007 DUP LS-05 DUP	9.83E-3 ± 16%	1.38E-4 ± 13%	6.28E-4 ± 6%	8.84E-3 ± 2%	7.57E-4 ± 5%	4.90E-5 ± 22%	1.04E-2 ± 2%	2
RPD MD	13%	16%	20% 1.40	2%	34% 3.15	2%	6%	
01-1008 PB Process Blank	2.63E-4 ± 9%	<4.E-7	2.83E-5 ± 5%	1.68E-4 ± 2%	7.30E-5 ± 3%	<4.E-7	2.70E-4 ± 2%	3
01-1008 LS-06	1.87E-2 ± 11%	1.93E-4 ± 11%	9.50E-4 ± 5%	1.30E-2 ± 2%	9.65E-4 ± 5%	7.27E-5 ± 19%	1.52E-2 ± 2%	3
01-1008 DUP LS-06 DUP	1.24E-2 ± 12%	1.92E-4 ± 11%	8.66E-4 ± 5%	1.14E-2 ± 2%	8.26E-4 ± 5%	7.26E-5 ± 18%	1.34E-2 ± 2%	3
RPD MD	41% 1.24	1%	9%	13%	16%	0%	13%	
01-1009 LS-07	1.43E-2 ± 14%	1.81E-4 ± 12%	1.03E-3 ± 5%	1.34E-2 ± 2%	1.18E-3 ± 5%	8.09E-5 ± 18%	1.59E-2 ± 2%	3
01-1009 DUP LS-07 DUP	1.41E-2 ± 12%	1.70E-4 ± 12%	1.00E-3 ± 5%	1.25E-2 ± 2%	9.82E-4 ± 5%	4.54E-5 ± 24%	1.47E-2 ± 2%	3
RPD MD	1%	6%	3%	7%	18%	56% 0.98	8%	
01-1010 LS-08	1.82E-2 ± 11%	2.41E-4 ± 11%	1.10E-3 ± 5%	1.32E-2 ± 2%	9.65E-4 ± 6%	1.09E-4 ± 17%	1.56E-2 ± 2%	3
01-1010 DUP LS-08 DUP	1.29E-2 ± 13%	1.98E-4 ± 13%	9.70E-4 ± 6%	1.29E-2 ± 2%	1.00E-3 ± 6%	9.26E-5 ± 19%	1.52E-2 ± 2%	3
RPD MD	34% 1.01	20%	13%	2%	4%	16%	3%	

Measured Activities (µCi/g) with 1-sigma error

ALO ID Client ID	Total Alpha ± 1s	Alpha Energy Analysis: U-234+ Np-237 ± 1s	Pu-239+ Pu-240 ± 1s	Pu-238+ Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of alpha emitters	Hot Cell Batch
01-1011 LS-09	1.35E-2 ± 13%	1.86E-4 ± 11%	9.00E-4 ± 5%	1.13E-2 ± 2%	8.53E-4 ± 5%	8.06E-5 ± 17%	1.33E-2 ± 2%	3
01-1011 DUP LS-09 DUP	1.55E-2 ± 11%	1.83E-4 ± 8%	1.07E-3 ± 3%	1.17E-2 ± 2%	8.70E-4 ± 4%	5.32E-5 ± 15%	1.39E-2 ± 2%	3
RPD MD	14%	2%	17%	3%	2%	41% 0.86	4%	
01-1012 PB Process Blank	2.25E-4 ± 9%	<5.E-7	2.61E-5 ± 5%	1.67E-4 ± 2%	6.17E-5 ± 3%	<5.E-7	2.56E-4 ± 2%	4
01-1012 LS-10	1.94E-2 ± 10%	2.43E-4 ± 11%	1.05E-3 ± 5%	1.19E-2 ± 2%	1.02E-3 ± 5%	6.90E-5 ± 21%	1.43E-2 ± 2%	4
01-1012 DUP LS-10 DUP	1.51E-2 ± 13%	2.02E-4 ± 11%	1.12E-3 ± 5%	1.31E-2 ± 2%	9.36E-4 ± 5%	7.84E-5 ± 18%	1.54E-2 ± 2%	4
RPD MD	25% 0.78	18%	6%	10%	9%	13%	8%	
01-1012 Lab Dup LS-10 DUP	N/A	2.11E-4 ± 11%	1.07E-3 ± 5%	1.22E-2 ± 2%	9.69E-4 ± 5%	6.62E-5 ± 20%	1.45E-2 ± 2%	4
01-1013 LS-11	1.44E-2 ± 12%	1.87E-4 ± 11%	1.01E-3 ± 5%	1.13E-2 ± 2%	8.70E-4 ± 5%	5.53E-5 ± 21%	1.34E-2 ± 2%	4
01-1013 DUP LS-11 DUP	1.63E-2 ± 12%	1.89E-4 ± 10%	1.07E-3 ± 4%	1.17E-2 ± 2%	8.82E-4 ± 5%	7.23E-5 ± 17%	1.39E-2 ± 2%	4
RPD MD	12%	1%	6%	3%	1%	27% 0.50	4%	
01-1014 PB Process Blank	1.78E-3 ± 3%	<1.E-6	3.41E-4 ± 2%	2.23E-3 ± 2%	2.01E-3 ± 2%	2.79E-6 ± 24%	4.58E-3 ± 1%	1
01-1014 LS-12	1.18E-2 ± 15%	1.71E-4 ± 11%	6.64E-4 ± 6%	8.68E-3 ± 2%	5.29E-4 ± 6%	3.54E-5 ± 25%	1.01E-2 ± 2%	1
01-1014 Dup LS-12 DUP	1.03E-2 ± 15%	1.79E-4 ± 11%	7.56E-4 ± 5%	8.54E-3 ± 2%	5.47E-4 ± 6%	3.17E-5 ± 27%	1.01E-2 ± 2%	1
RPD	14%	5%	13%	2%	3%	11%	0%	
01-1015 LS-13	7.90E-3 ± 17%	1.88E-4 ± 11%	7.32E-4 ± 5%	8.48E-3 ± 2%	6.10E-4 ± 6%	3.93E-5 ± 24%	1.00E-2 ± 2%	1

Measured Activities (µCi/g) with 1-sigma error

ALO ID Client ID	Total Alpha ± 1s	Alpha Energy Analysis U-234+ Np-237 ± 1s	Pu-239+ Pu-240 ± 1s	Pu-238+ Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of alpha emitters	Hot Cell Batch
01-1016 LS-14	<4.E-3	3.69E-5 ± 24%	1.20E-4 ± 13%	1.32E-3 ± 4%	1.07E-4 ± 14%	<2.E-5	1.60E-3 ± 4%	1
01-1017 PB Process Blank	6.31E-3 ± 9%	<2.E-5	9.48E-4 ± 5%	4.82E-3 ± 2%	1.75E-3 ± 3%	<2.E-5	7.56E-3 ± 2%	5
01-1017 LS-16	4.70E+0 ± 3%	<1.E-3	2.52E-1 ± 3%	4.37E+0 ± 2%	1.70E-1 ± 3%	1.58E-2 ± 10%	4.81E+0 ± 2%	5
01-1017 DUP LS-16 DUP	4.16E+0 ± 3%	8.33E-3 ± 28%	2.25E-1 ± 5%	3.70E+0 ± 2%	1.52E-1 ± 6%	1.31E-2 ± 22%	4.10E+0 ± 2%	5
RPD	12%		11%	17%	11%	19%	16%	
MS-1003	105%							
MS-1006			95%					
MS-1012			104%					
BS-1	102%		113%					
BS-2			116%					
Blank 1	<4.E-3	<2.E-5	<2.E-5	<3.E-5	<2.E-5	<2.E-5		
Blank 2		<2.E-5	<2.E-5	<2.E-5	<9.E-6	<9.E-6		

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

11/14/02

Client : R. Hallen

Rev. 1

Cognizant Scientist: L R Greenwood Date : 11/19/02

Concur : C Soderquist Date : 11-25-02

Measured Activities (µCi/g) with 1-sigma error

ALO ID / Client ID	Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of Am + Cm	Hot Cell Batch
	Americium/Curium Analysis				
01-1014 PB	3.07E-4	7.72E-4	<8.E-7	1.08E-3	1
Process Blank	± 3%	± 2%		± 2%	
01-1014	8.49E-3	5.48E-4	5.06E-5	9.09E-3	1
LS-12	± 2%	± 7%	± 21%	± 2%	
01-1014 Dup	8.39E-3	5.42E-4	6.23E-5	8.99E-3	1
LS-12 DUP	± 2%	± 6%	± 18%	± 2%	
RPD	1%	1%	21%	1%	
MD			0.38		
01-1014 Lab DUP	8.18E-3	5.32E-4	5.95E-5	8.77E-3	1
LS-12	± 3%	± 8%	± 24%	± 3%	
01-1015	8.30E-3	5.57E-4	3.59E-5	8.89E-3	1
LS-13	± 2%	± 6%	± 25%	± 2%	
01-1016	1.17E-3	8.36E-5	6.69E-6	1.26E-3	1
LS-14	± 3%	± 9%	± 33%	± 3%	
01-1017 PB	2.84E-3	1.84E-3	<3.E-5	4.71E-3	2
Process Blank	± 4%	± 5%		± 3%	
01-1017	3.99E+0	1.64E-1	1.09E-2	4.16E+0	2
LS-16	± 2%	± 6%	± 21%	± 2%	
01-1017 DUP	3.45E+0	1.37E-1	1.08E-2	3.60E+0	2
LS-16 DUP	± 3%	± 7%	± 24%	± 3%	
RPD	15%	18%	1%	15%	
MS-1014	100%				
BS-1003	99%				
BLK-1003	<2.E-5	<2.E-5	<7.E-6		

The samples were prepared in the hot cells in two batches.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

11/14/02

Client : R. Hallen

Rev. 1

Cognizant Scientist: LRGreenwood Date : 11/14/02

Concur : C Soderquist Date : 11-25-02

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Tc-99 Error +/-
01-1017 PB Process Blank	<4.E-4
01-1017 LS-16	1.03E-1 ± 4%
01-1017 DUP LS-16 DUP	8.17E-2 ± 4%
RPD MD	23% 2.03
MS-1203	79%
BS-1003	95%
Blk-1017	<6.E-7

Battelle PNNL/RSE/Inorganic Analysis ... ICPAES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#: 42365 / W57984
ASR#: 6107
Client: R. Hallen
Total Samples: 14 liquids

RPL#:	01-01003	01-01016
Client ID:	"LS-01"	"LS-14"
Sample Preparation: PNL-ALO-128 (1mL/26mL or 1.2g/26mL)		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File):
05-25-2001 (A0676)
06-15-2001 (A0682)
06-19-2001 (A0684)
06-20-2001 (A0885)

See **Chemical Measurement Center 98620 file:** ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number:
WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

9-20-01
Reviewed by

9-25-01
Concur

Fourteen liquid samples from Analytical Service Request (ASR) 6107 were prepared by acid digestion per PNL-ALO-128 in the Shielded Analytical Laboratory (SAL). The samples were prepared by using nominal 1.0 mL of sample and diluting to a final volume of about 26 mL. The final volume was calculated by using the mass and density of the resulting digestate.

In the ASR, Al, Ba, Ca, Cd, Cr, Fe, K, La, Mg, Mn, Na, Ni, P, Pb, and Sr were identified as analytes of interest for this work. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those detected as part of the ICPAES analysis are reported, but have concentrations less than the method detection limit (MDL) and have not been fully evaluated for QC performance.

The attached ICPAES Results (9 pages; 2 pages from each of the analysis runs on 5-25, 6-19, and 6-20, and 3 pages from analysis run on 6-15) presents the final results. Results are from the direct measurement of the digestates, except for Na which required an additional 5x dilution to bring the Na concentration within the ICPAES linear range. The ICPAES measurement results are reported in µg/g of liquid sample (as requested by the ASR) and have been corrected for all dilutions resulting from sample processing. It should be noted that the preliminary results reported were presented on a µg/mL basis.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. A digestion processing blank, laboratory control sample (blank spike), matrix spike, and duplicate were prepared with the sample for each processing batch. The blank spikes was prepared by using 3 mL of a custom multi-element solutions "010514i901 and 010514i902" per 26 mL digestate volume, and the matrix spikes were prepared by using 1 mL of the same multi-element solutions.

Process Blank:

Concentration of analytes of interest measured in the three process blanks were all within acceptance criteria of ≤ EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

Blank Spike (laboratory control sample):

The blank spike recovery for analytes of interest was within the acceptance criteria of 80% to 120%, except for Na for the 6-19-2001 analysis which recovered slightly high at 121%.

Duplicate RPD (Relative Percent Difference):

For those analytes of interest measured above the EQL, the RPDs were within the acceptance criteria of less than 20%. Even for analytes with concentration between the method detection limit (MDL) and the EQL, the RPDs are quite good, with only the Mg for LS-06 exceeding the 20% criteria.

Matrix Spiked Sample:

Matrix spike were prepared for LS-02, LS-06, LS-10, and LS-12. Except for Al and Na, which had spike concentration less than 20% of the sample concentration, the analytes of interest meet the matrix spike recovery criteria of 75% to 125%. Post spiking or serial dilution is required for the Al and Na.

Post-Spiked Samples (Spike A Elements):

Post spiking was performed on LS-02, LS-06 and LS-10. All post-spiked analytes of interest in samples tested were recovered within tolerance of 75% to 125%, except Al and Na. The post spike analysis uses a general spiking solution intended to be usable on the majority of sample analyzed by ICPAES. However, for the sample selected for post spiking, the spike concentration for Al and Na was less than 20% of the sample concentration and the recovery results are considered meaningless. For these analytes, the use of serial dilution results is required to evaluate potential matrix interferences.

Post-Spiked Samples (Spike B Elements):

The post spike recovery for La, which is the only Spike B analyte of interest, were within tolerance of 75% to 125%.

Serial dilution:

Serial dilution was required for Al, Na, Sr, since for these analytes the post spike concentrations were less than 20% of the sample concentration (i.e., recoveries could not be evaluated). These analytes demonstrated a percent difference (%D) within the acceptance criteria of ±10% after correcting for dilution for all the serial dilutions measured. In some case the samples were not have enough serial dilutions to be able to calculate the Na %Diff (i.e., at all dilutions except for the highest dilution, the Na was over range).

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (Det. Limit) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' times the Detection Limit.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RSE/Inorganic Analysis... ICPAES Results

	Run Date=	5/25/2001	5/25/2001	5/25/2001	5/25/2001	5/25/2001	5/25/2001	5/25/2001
	Multiplier=	20.6	103.6	512.8	103.4	103.9	102.8	24.3
	RPL#=	01-1014-PB	01-1003	01-1003-D	01-1014	01-1014-D	01-1015	01-1016
Det. Limit	Client ID=	Process Blank (ALO-128)	LS-01	LS-01-Dup	LS-12	LS-12-Dup	LS-13	LS-14
ug/mL	Analytes	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.060	Al	[4.0]	7,230	7,320	6,960	6,930	6,800	1,180
0.010	Ba	--	--	--	--	--	--	--
0.250	Ca	--	[160]	[150]	[130]	[120]	[120]	[34]
0.015	Cd	--	22.7	[23]	22.9	22.2	22.1	3.96
0.020	Cr	--	89.2	[89]	94.0	93.2	91.2	15.8
0.025	Fe	--	[7.3]	--	[3.2]	[3.2]	[3.3]	[1.3]
2.000	K	--	[720]	--	[710]	[680]	[690]	[130]
0.050	La	--	--	--	--	--	--	--
0.100	Mg	--	--	--	--	--	--	--
0.050	Mn	--	--	--	--	--	--	--
0.150	Na	48.2	95,700	99,400	91,100	91,400	90,200	22,900
0.030	Ni	--	165	167	163	162	158	27.8
0.100	P	--	685	670	667	656	638	99.3
0.100	Pb	--	[61]	--	[55]	[52]	[51]	[13]
0.015	Sr	--	--	--	74.5	74.5	73.0	15.2
	Other Analytes							
0.025	Ag	--	--	--	--	--	--	--
0.250	As	--	--	--	--	--	--	--
0.050	B	39.4	63.1	[60]	58.8	59.3	57.5	87.0
0.010	Be	--	--	--	--	--	--	--
0.100	Bi	--	--	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--	--
0.050	Co	--	--	--	--	--	--	--
0.025	Cu	--	[3.7]	--	[6.7]	[6.1]	[6.0]	[0.94]
0.050	Dy	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--
0.030	Li	--	--	--	--	--	--	--
0.050	Mo	--	[19]	--	[20]	[19]	[19]	[5.0]
0.100	Nd	--	--	--	--	--	--	--
0.750	Pd	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--
0.500	Si	[81]	[160]	--	[160]	[160]	[160]	669
1.500	Sn	--	--	--	--	--	--	--
1.500	Te	--	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--	--
0.025	Ti	--	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--	--
2.000	U	--	--	--	--	--	--	--
0.050	V	--	--	--	--	--	--	--
2.000	W	--	--	--	--	--	--	--
0.050	Y	--	--	--	--	--	--	--
0.050	Zn	--	--	--	[5.5]	[5.4]	[5.5]	[2.2]
0.050	Zr	--	--	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RSE/Inorganic Analysis... ICPAES Results

QC Performance 5/25/2001

Criteria>	<20%	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%	< +/-10%	< +/-10%	< +/-10%
QC ID=	01-1003 & 01-1003D	01-1014 & 01-1014D	010514i901 010514i902 LCS/BS	01-1014 MS	01-1014 + Post Spike A	01-1014 + Post Spike B	01-1003 @5/@25 Serial Dil	01-1014 @5/@25 Serial Dil	01-1015 @5/@25 Serial Dil	01-1016 @1/@5 Serial Dil
Analytes	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff	%Diff	%Diff	%Diff
Al	3.4	0.0	96.7	n.r.	n.r.		-0.5	-0.2	-0.9	2.0
Ba			95.6	98.8	102.8					
Ca	1.1	0.4	95.6	102.2	104.1					
Cd	2.1	2.7	95.2	100.2	103.1					
Cr	2.3	0.5	96.0	101.8	105.4					
Fe		1.8	98.5	104.0	108.1					
K		4.0	93.3	98.5	97.9					
La			92.7	98.4		98.4				
Mg			98.2	106.6	111.3					
Mn			98.8	106.9	109.7					
Na	6.0	0.9	97.4	n.r.	n.r.		-2.4	-1.7	-2.8	4.1
Ni	3.3	0.3	97.6	106.3	108.4					
P	0.1	1.3	95.0	101.9	102.1					
Pb		3.4	97.7	103.1	105.8					
Sr		0.5	95.7	106.3	103.0					
Other Analytes										
Ag			91.2	97.3	100.4					
As					105.3					
B	3.3	1.2			106.5					
Be					104.0					
Bi			95.4	100.1	103.6					
Ce						99.1				
Co					109.5					
Cu		8.3	97.7	100.9	107.4					
Dy						101.7				
Eu						109.0				
Li					102.2					
Mo		1.0			105.2					
Nd			92.5	96.1		98.2				
Pd			93.9	93.0		89.7				
Rh			94.7	98.3		97.4				
Ru			91.5							
Sb					102.5					
Se					108.4					
Si		4.1	124.5	105.8	121.4					
Sn										
Te										
Th						102.3				
Ti			92.9	96.2	101.1					
Tl					104.0					
U			92.4	93.9		96.0				
V					99.5					
W										
Y					100.7					
Zn		1.8	96.6	102.0	105.8					
Zr			96.2	102.0	105.2					

n.r. = not recovered; spike is less than 20% of sample concentration.

Battelle PNNL/RSE/Inorganic Analysis.... ICPAES Results

	Run Date=	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001
	Multiplier=	1.0	19.6 (Na98.2)	20.3 (Na101.7)	20.1 (Na100.3)	20.3 (Na101.3)	20.0 (Na100.0)	20.1 (Na100.6)	19.9 (Na99.6)	19.1 (Na95.6)
	RPL#=	01-1004-B	01-1004	01-1004-D	01-1005	01-1005-D	01-1006	01-1006-D	01-1007	01-1007-D
Det. Limit	Client ID=	Process Blank (ALO-128)	LS-02	LS-02 Dup	LS-03	LS-03 Dup	LS-04	LS-04 Dup	LS-05	LS-05 Dup
ug/mL	Analytes	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.060	Al	17.5	6,710	6,750	6,500	6,570	6,800	6,640	6,750	6,830
0.010	Ba	--	--	--	--	--	--	--	--	--
0.250	Ca	[5.9]	119	121	105	108	117	113	114	116
0.015	Cd	--	20.9	21.0	20.1	20.3	21.0	20.7	20.9	21.1
0.020	Cr	--	76.7	77.5	80.4	80.8	84.0	82.3	83.9	85.0
0.025	Fe	[1.1]	[4.7]	[4.6]	[2.6]	[2.4]	[2.6]	[2.6]	[2.6]	[2.4]
2.000	K	--	814	824	809	816	841	815	817	828
0.050	La	--	[2.2]	[2.3]	--	--	--	--	--	--
0.100	Mg	--	--	--	--	[2.2]	[2.3]	--	--	--
0.050	Mn	--	--	--	--	--	--	--	--	--
0.150	Na	61.1	89,900	90,900	86,200	87,600	91,000	90,200	91,500	92,000
0.030	Ni	--	145	147	143	143	148	145	148	149
0.100	P	--	631	638	624	628	647	634	641	648
0.100	Pb	--	57.5	58.4	42.5	42.2	46.2	45.3	46.0	46.4
0.015	Sr	--	161	168	100	102	95.5	91.0	84.9	83.5
	Other Analytes									
0.025	Ag	--	--	--	--	--	--	--	--	--
0.250	As	--	--	--	--	--	--	--	--	--
0.050	B	27.9	64.4	75.5	60.1	93.5	95.3	58.7	53.5	65.1
0.010	Be	--	--	--	--	--	--	--	--	--
0.100	Bi	--	[2.4]	[2.2]	--	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--	--	--	--
0.050	Co	--	[1.5]	[1.5]	[1.5]	[1.5]	[1.5]	[1.5]	[1.5]	[1.5]
0.025	Cu	--	[4.3]	[4.3]	[4.2]	[4.2]	[4.4]	[4.2]	[4.3]	[4.3]
0.050	Dy	--	--	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--	--	--
0.030	Li	--	--	--	--	--	--	--	--	--
0.050	Mo	--	17.6	17.7	17.3	17.4	18.1	17.7	18.1	18.2
0.100	Nd	--	[6.8]	[7.0]	[3.5]	[3.4]	[3.2]	[3.1]	[2.9]	[2.9]
0.750	Pd	--	--	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--	--	--
0.500	Si	103	243	259	217	295	372	228	192	213
1.500	Sn	--	--	--	--	--	--	--	--	--
1.500	Te	--	--	--	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--	--	--	--
0.025	Ti	--	--	--	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--	--	--	--
2.000	U	--	--	--	--	--	--	--	--	--
0.050	V	--	--	--	--	--	--	--	--	--
2.000	W	--	--	--	--	--	--	--	--	--
0.050	Y	--	--	--	--	--	--	--	--	--
0.050	Zn	[1.1]	[2.2]	[2.0]	[1.4]	[1.1]	[1.1]	[1.5]	[1.4]	[1.3]
0.050	Zr	--	[2.0]	[2.0]	--	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 6/15/2001 - RPDs and LCS/BS Recovery

Criteria>	<20%	<20%	<20%	<20%	80% - 120%
QC ID=	01-1004 & 01-1004D	01-1005& 01-1005D	01-1006 & 01-1006D	01-1007 & 01-1007D	010514i901 010514i902 LCS/BS
Analytes	RPD (%)	RPD (%)	RPD (%)	RPD (%)	%Rec
Al	3.9	1.0	1.0	2.8	112.0
Ba					99.0
Ca	2.7	3.3	2.2	3.8	99.7
Cd	3.9	0.8	0.1	2.5	99.2
Cr	3.5	0.6	0.6	2.9	99.2
Fe	**7.3**	**4.9**	**2.9**	**8.0**	101.2
K	3.4	0.9	1.8	3.0	101.7
La	**0.5**				98.4
Mg					101.2
Mn					102.4
Na	1.1	1.6	0.9	0.5	119.2
Ni	3.4	0.6	0.5	2.5	99.5
P	3.4	0.7	0.6	2.6	98.0
Pb	2.9	0.6	0.6	2.6	100.6
Sr	0.6	1.9	3.4	0.1	99.0
Other Analytes					
Ag					**59.3**
As					
B	11.4	43.5	46.2	21.2	
Be					
Bi	**13.9**				98.9
Ce					
Co	**0.5**	**2.0**	**0.2**	**1.6**	
Cu	4.2	0.7	2.5	3.5	100.0
Dy					
Eu					
Li					
Mo	3.5	0.4	0.8	2.6	
Nd	**2.0**	**3.6**	**0.0**	**0.1**	
Pd					99.5
Rh					100.2
Ru					96.4
Sb					
Se					
Si	2.0	30.5	46.8	12.2	**159.1**
Sn					
Te					
Th					
Ti					95.9
Tl					
U					99.0
V					
W					
Y					
Zn	**16.3**	**22.9**	**30.7**	**0.9**	98.2
Zr	**2.0**				98.7

Shaded results exceed acceptance criteria

Bold and unshaded RPDs indicate one or both results <EQL.

QC Performance 6/15/2001- MS & PS Recovery and %Diff

Criteria>	75%-125%	75%-125%	75%-125%	< +/-10%	< +/-10%	< +/-10%	< +/-10%
QC ID=	01-1004 MS	01-1004 + Post Spike A	01-1004 + Post Spike B	01-1004 @1/@5 Serial Dil	01-1005 @1/@5 Serial Dil	01-1006 @1/@5 Serial Dil	01-1007 @1/@5 Serial Dil
Analytes	%Rec	%Rec	%Rec	%Diff	%Diff	%Diff	%Diff
Al	n.r.	n.r.		3.7	1.4	2.2	2.7
Ba	93.0	95.3					
Ca	98.5	97.7					
Cd	96.7	99.6					
Cr	91.5	99.1					
Fe	97.2	99.3					
K	96.1	96.0					
La	95.6		97.0				
Mg	99.3	103.4					
Mn	103.4	103.3					
Na	n.r.	n.r.		n.m.	n.m.	n.m.	n.m.
Ni	88.5	99.0					
P	95.5	96.4					
Pb	97.8	100.3					
Sr	n.r.	99.3					
Other Analytes							
Ag	95.7	96.1					
As		100.2					
B		99.6					
Be		97.6					
Bi	96.6	97.7					
Ce			98.7				
Co		101.1					
Cu	96.8	100.0					
Dy			98.5				
Eu			105.7				
Li		98.2					
Mo		98.0					
Nd			99.8				
Pd	103.0		91.1				
Rh	101.6		96.2				
Ru	105.0						
Sb		97.8					
Se		99.9					
Si	120.1	107.2					
Sn							
Te							
Th			98.1				
Ti	91.1	93.2					
Tl		95.4					
U	95.9		96.6				
V		94.4					
W							
Y		96.0					
Zn	98.6	102.1					
Zr	97.4	97.6					

Shaded results exceed acceptance criteria

n.r. = not recovered; spike concentration less than 20% of sample concentration

n.m. = not measured; insufficient dilutions prepared to evaluate %Diff.

Battelle PNNL/RSE/Inorganic Analysis.... ICPAES Results

	Run Date=	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001
	Multiplier=	20.3	20.1 (Na100.3)	20.1 (Na100.6)	21.1 (Na105.4)	20.5 (Na102.6)	20.8 (Na103.9)	20.5 (Na102.3)	19.9 (Na99.4)	19.4 (Na97.2)
	RPL#=	01-1008-B	01-1008	01-1008-D	01-1009	01-1009-D	01-1010	01-1010-D	01-1011	01-1011-D
Det. Limit	Client ID=	Proc. Blk	LS-06	LS-06	LS-07	LS-07	LS-08	LS-08	LS-09	LS-09
ug/mL	Analytes	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.060	Al	[4.6]	7,180	6,990	6,640	6,670	6,840	6,960	6,610	6,600
0.010	Ba	[0.22]	[0.23]	--	--	[0.22]	--	[0.23]	--	[0.27]
0.250	Ca	--	127	122	120	123	122	125	122	120
0.015	Cd	--	22.4	21.8	21.1	20.9	21.4	22.0	20.8	20.6
0.020	Cr	--	90.3	88.5	85.2	84.3	87.0	88.6	84.5	84.2
0.025	Fe	[0.65]	[3.2]	[2.7]	[4.6]	[4.7]	[5.0]	5.83	[4.8]	[4.8]
2.000	K	--	895	864	817	794	798	812	769	768
0.050	La	--	--	--	[1.2]	--	--	--	--	--
0.100	Mg	--	[2.7]	[2.1]	--	[2.1]	--	--	[2.1]	--
0.050	Mn	--	--	--	--	--	--	--	--	--
0.150	Na	46.5	98,700	97,200	92,500	92,500	94,500	97,600	93,800	94,200
0.030	Ni	--	159	156	149	147	152	154	147	147
0.100	P	--	665	649	629	622	610	614	622	620
0.100	Pb	--	49.4	48.3	46.5	43.7	45.8	46.4	44.4	44.2
0.015	Sr	--	87.6	86.3	185	185	174	178	160	161
	Other Analytes									
0.025	Ag	--	--	--	--	--	--	--	--	--
0.250	As	--	--	--	--	--	--	--	--	--
0.050	B	36.7	104	52.4	43.5	88.0	43.4	69.0	70.7	65.0
0.010	Be	--	--	--	--	--	--	--	--	--
0.100	Bi	--	[2.9]	[2.6]	[2.6]	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--	--	--	--
0.050	Co	--	[1.7]	[1.7]	[1.6]	[1.4]	[1.5]	[1.5]	[1.4]	[1.4]
0.025	Cu	--	[4.7]	[4.5]	[4.4]	[4.2]	[4.4]	[4.5]	[4.3]	[4.3]
0.050	Dy	--	--	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--	--	--
0.030	Li	--	[0.61]	[0.68]	--	--	--	--	--	--
0.050	Mo	--	19.0	18.8	17.9	17.5	18.1	18.5	17.6	17.6
0.100	Nd	--	[4.4]	[4.5]	[4.8]	[3.4]	[3.4]	[3.5]	[3.2]	[3.1]
0.750	Pd	--	--	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--	--	--
0.500	Si	[86]	385	206	199	317	220	268	300	245
1.500	Sn	--	--	--	--	--	--	--	--	--
1.500	Te	--	--	--	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--	--	--	--
0.025	Ti	--	--	--	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--	--	--	--
2.000	U	--	--	[40]	--	--	--	--	--	--
0.050	V	--	--	--	--	--	--	--	--	--
2.000	W	--	--	--	--	--	--	--	--	--
0.050	Y	--	--	--	--	--	--	--	--	--
0.050	Zn	[1.1]	[1.6]	[1.7]	[3.9]	[3.4]	[4.2]	[4.2]	[4.3]	[4.5]
0.050	Zr	--	[1.2]	[1.1]	[1.1]	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 6/19/2001

Criteria>	<20%	<20%	<20%	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	01-1008 & 01-1008D	01-1009 & 01-1009D	01-1010 & 01-1010D	01-1011 & 01-1011D	010514i901 010514i902 LCS/BS	01-1008 MS	01-1008 + Post Spike A	01-1008 + Post Spike B	01-1008 @1/@5 Serial Dil
Analytes	RPD (%)	RPD (%)	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	2.8	0.5	1.7	0.1	111.0	n.r.	n.r.		2.8
Ba					99.2	92.6	101.9		
Ca	4.3	2.0	2.6	1.3	99.7	96.7	102.5		
Cd	2.5	0.7	2.4	0.8	97.9	95.1	103.3		
Cr	2.0	1.1	1.8	0.3	97.8	89.8	104.5		
Fe	**17.6**	**1.9**	14.9	**0.5**	101.3	96.6	105.0		
K	3.6	2.8	1.8	0.0	99.6	95.4	100.6		
La					98.2	97.6		100.5	
Mg	**25.1**				100.9	97.5	108.4		
Mn					100.9	101.1	107.4		
Na	1.5	0.0	3.2	0.4	121.1	n.r.	n.r.		n.m.
Ni	1.9	1.4	1.5	0.1	99.9	84.2	109.4		
P	2.4	1.1	0.6	0.4	96.1	91.7	102.0		
Pb	2.3	6.2	1.3	0.5	102.3	99.4	107.8		
Sr	1.5	0.1	2.0	0.4	99.4	94.1	104.3		
Other Analytes									
Ag					55.3	94.1	99.1		
As							103.6		
B	**66.2**	**67.7**	**45.5**	**8.3**			105.3		
Be							100.9		
Bi	10.3				96.0	93.4	99.8		
Ce								102.7	
Co	**0.3**	**12.6**	**0.7**	**0.7**			107.6		
Cu	**5.6**	**4.5**	**1.7**	**0.3**	99.4	95.8	105.3		
Dy								102.0	
Eu								109.7	
Li	10.8						102.3		
Mo	1.4	2.5	1.9	0.3			103.8		
Nd	**3.0**	**33.6**	**1.2**	**1.7**				102.9	
Pd					97.0	97.6		88.2	
Rh					97.8	99.8		100.8	
Ru					96.0	104.6			
Sb							101.5		
Se							104.4		
Si	**60.5**	**45.8**	**19.6**	**20.0**	**154.8**	96.9	114.4		
Sn									
Te									
Th								103.1	
Ti					96.0	91.4	99.0		
Tl							101.2		
U					96.9	94.4		99.0	
V							98.7		
W									
Y							98.5		
Zn	**3.8**	**13.8**	**0.4**	**2.8**	99.5	100.4	108.4		
Zr	**3.4**				98.9	98.1	103.6		

Shaded results do not meet QC acceptance criteria.

n.r. = not recovered; spike is less than 20% of sample concentration.

Bold and unshaded RPDs indicate one or both results <EQL.

n.m. = not measured; insufficient dilutions prepared to evaluate %Diff.

	Run Date=	6/20/2001	6/20/2001	6/20/2001	6/20/2001	6/20/2001
	Multiplier=	1.0	21.3 (Na106.7)	20.3 (Na101.4)	21.1 (Na105.5)	21.2 (Na105.8)
	RPL#=	01-1012-B	01-1012	01-1012-D	01-1013	01-1013-D
Det. Limit	Client ID=		LS-10	LS-10	LS-11	LS-11
ug/mL	Analytes	ug/g	ug/g	ug/g	ug/g	ug/g
0.060	Al	[0.17]	6,580	6,540	6,590	6,600
0.010	Ba	[0.011]	--	--	[0.21]	--
0.250	Ca	--	118	117	120	120
0.015	Cd	[0.016]	21.2	21.1	21.2	21.2
0.020	Cr	--	86.1	85.1	88.1	88.7
0.025	Fe	[0.028]	[4.8]	[4.9]	[4.6]	[4.7]
2.000	K	--	795	789	784	780
0.050	La	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.150	Na	2.23	90,100	88,500	90,600	90,700
0.030	Ni	--	147	146	149	149
0.100	P	--	616	614	586	587
0.100	Pb	--	46.9	46.2	47.2	47.3
0.015	Sr	--	129	128	113	113
	Other Analytes					
0.025	Ag	--	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	1.68	54.8	52.6	56.5	52.9
0.010	Be	--	--	--	--	--
0.100	Bi	[0.15]	[3.5]	[2.3]	[2.3]	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	[1.5]	[1.6]	[1.6]	[1.6]
0.025	Cu	--	5.51	5.40	6.46	6.39
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.050	Mo	--	17.6	17.4	17.6	17.8
0.100	Nd	--	[3.9]	[4.1]	[3.8]	[3.9]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[3.1]	208	189	202	185
1.500	Sn	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[0.054]	[5.0]	[5.0]	[5.3]	[5.2]
0.050	Zr	--	[1.1]	[1.1]	[1.1]	[1.1]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 6/20/2001

Criteria>	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	01-1013 & 01-1013 D	01-1012-BS	01-1012 MS	none + Post Spike A	none + Post Spike B	01-1013 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	0.0	106.3	n.r.			2.8
Ba		110.1	86.7			
Ca	0.0	111.7	90.3			
Cd	0.1	111.4	87.8			
Cr	0.2	112.6	79.9			
Fe	7.3	115.6	90.5			
K	3.5	111.2	87.3			
La			90.1			
Mg		118.7	92.5			
Mn		116.1	93.0			
Na	0.2	101.7	n.r.			n.m.
Ni	0.1	117.7	78.1			
P	0.1	110.0	80.4			
Pb	2.8	116.0	89.6			
Sr	0.0	111.3	n.r.			3.7
Other Analytes						
Ag		107.1	88.4			
As		109.2				
B	5.9	n.r.				3.4
Be		108.5				
Bi		111.0	87.5			
Ce						
Co		114.5				
Cu	2.7	114.0	88.5			
Dy						
Eu						
Li		114.9				
Mo	0.7					4.7
Nd						
Pd			90.6			
Rh			92.2			
Ru			96.0			
Sb						
Se		109.4				
Si	6.3		97.7			
Sn						
Te						
Th						
Ti			85.0			
Tl		108.8				
U			89.3			
V		105.8				
W						
Y		106.4				
Zn	0.5	119.0	91.6			
Zr			91.6			

Shaded results exceed acceptance criteria

Bold results for information only; LCS or Serical Dilution concentration less than EQL.

n.r. = not recovered; spike concentration less than 20% of sample concentration

n/a = not applicable; KOH flux and Ni crucible used for preparing samples.

Battelle Pacific Northwest National Laboratory
Radiochemical Processing Group
Shielded Facility Operations Team

WT% SOLIDS DATA SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

CLIENT: Hallen/Smith | WORK PACKAGE: W57984 – Mult (Smith) | ASR/ARF/LOI/TI: 6107

QA PLAN: SBMS | IMPACT LEVEL: N/A | PROCEDURE NO.: PNL-ALO-504

Sr/TRU Washed Solids- HLW Feed
SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	TARE WEIGHT (g)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	WEIGHT % SOLIDS
01-01017	LS-16	8.6983	12.5691	10.3950, 10.3934	43.79
01-01017-D	LS-16-Duplicate	8.2702	12.2389	10.3130, 10.3100	51.40

$$\text{WT\% SOLIDS} = \frac{B - TARE}{A - TARE} \times 100$$

DATE/TIME IN: 5/~~15~~14/01 2:30 | OVEN TEMPERATURE: 105 °C

DATE/TIME OUT: 5/16/01 8:15 | OVEN TEMPERATURE: 103 °C

BALANCE : CELL 2 (360-06-01-016) X

TEMPERATURE READOUT: 2115 | Expiration Date: 4/01

THERMOCOUPLE: 3118 | Expiration Date: 4/02

Analyst | Date 5/16/01 | Reviewer | Date 5/16/01

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client: R. Hallen
RPL Numbers: 01-01014 to 01-01017
Analyst: MJ Steele

Charge Code/Project: W57984 / 42365
ASR Number: 6107
Analysis Date: July 19/20/31, 2001

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"
M&TE: Carbon System (WA92040); Balance (360-06-01-023)

Analysis Results

RPL #	Liquid Sample ID	TIC ugC/mL	TIC RPD	TOC ugC/mL	TOC RPD	TC ugC/mL	TC RPD
01-01014	LS-12	8,540		12,000		20,500	
01-01014 Dup	LS-12	8,550	0%	12,200	2%	20,700	1%
01-01015	LS-13	7,930		11,500		19,400	
01-01015 Dup	LS-13	7,680	3%	11,400	1%	19,100	2%
01-01015 Trip	LS-13	8,700		12,100		20,800	
01-01016	LS-14	1,500		3,140		4,640	
01-01016 Dup	LS-14	1,500	0%	3,030	3%	4,540	2%
01-01016 MS	Recovery	101%		99%		100%	
BS/LCS (07-19-01)	Recovery	102%		106%			
BS/LCS (07-20-01)	Recovery	103%		102%			
RPL #	**Solid Sample ID**	**TIC ugC/g**	**TIC RPD**	**TOC ugC/g**	**TOC RPD**	**TC ugC/g**	**TC RPD**
01-01017	LS-16	18,500		6,190		24,700	
01-01017 Dup	LS-16	17,700	4%	6,960	12%	24,700	0%
01-01017 MS	Recovery	104%		90%		97%	
BS/LCS (07-31-01)	Recovery	101%		102%			

The TOC/TIC analyses of the samples submitted under ASRs 6107 are to be performed by both the hot persulfate and furnace methods. This report presents the results from the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-381.

Q.C. Comments:

The standards for TIC and TOC supernatant analysis are liquid carbon standards from VWR. The lot numbers and Chemical Management System (CMS) numbers for the standards are included on the raw data benchsheets.

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

For TIC and TOC solids analysis pure chemical compounds are used as the calibration, as well as matrix spiking, standards. The TIC analysis uses calcium carbonate and the TOC uses α-Glucose (JT Baker, Aldrich, Sigma, and Mallinckrodt lot numbers and CMS numbers are provided on the raw data benchsheets).

The QC for the methods involves calibration blanks, sample duplicates, laboratory control sample, and matrix spikes. The ASR indicates that the analyses are to be performed to "Conducting Analytical Work in Support of Regulatory Programs". The performance of the QC samples is compared to this QA Plan.

Laboratory Control Sample (LCS)/Blank Spike(BS): A LCS/BS was analyze each day that the samples were analyzed. The LCS/BSs for both the liquid analysis and the solids analysis were within acceptance criteria of 80% to 120%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spikes for the LS-14 liquid sample and the LS-16 solids sample demonstrate recoveries well within the acceptance criteria of 75% to 125% recovery.

Duplicates: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD) between sample and duplicate. The TIC and TOC RPD results are well within the acceptance criteria of <20% RPD.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL. The analysis MDLs (total ug C) are based on 3 times the standard deviation of a set of historical data. The sample MDLs (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by: [signature] Date 10-02-01

Review/Approval by: M. J. Steele Date 10/20/01

Excel Archive File: ASR 6014L 6031L 6107L&S 6121L.xls

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Hallen	Analyzer M&TE:	WC01713 -- 701
Project :	42365	Balance M&TE:	360-06-01-023
Work Pkg:	CMC	TOC STD: Glucose CSM-53219>>>	40.00% Carbon <<[G]
Analyzed:	July 31, 2001	TIC STD: CaCO3 CMS-139285>>>	11.99% Carbon <<[C]
ASR:	6107		

MJ Steele 10/20/01

		Raw TIC (ug C)	Raw TOC (ug C)	TIC	TOC	
				13.7	53.7	<<< Blank Average (ug C)
Blanks:	Calibration blank (start of batch)	13.0	59.0	1.2	11.0	<<< Blank Std Dev (ug C)
	Calibration blank (start of batch)	13.0	41.0	*2.16*	*5.8*	*<<< Pooled Std Dev (ug C)*
	Calibration blank (end of batch)	15.0	61.0	*6.5*	*17.3*	*<<< Method Det. Limit (ug C)*

Is Blank Std Dev < Method Det Limit?	
TIC	Yes
TOC	Yes

		Total Inorganic Carbon (TIC)					Total Organic Carbon (TOC)				
		[A] Raw TIC (ug)	[B] Blk (ug)	[D] Std wt (g)	TIC % Rec		[E] Raw TOC (ug)	[F] Blk (ug)	[H] Std wt (g)	TOC % Rec	
Standards:	Calibration Standard (start of batch)	912	14	0.0076	98.6		595	54	0.0014	96.7	
	Calibration Standard (start of batch)	897	14	0.0076	96.9		605	54	0.0014	98.5	
	Calibration Standard (end of batch)	834	14	0.0069	99.2		754	54	0.0019	92.1	
		[L] Average TIC % Rec >>>>			**98.2**	<<[L]	[P] Average TOC % Rec >>>>			**95.8**	<<[P]
QC	Laboratory Control Sample (BS)	2255	14	0.0188	101.2		1110	54	0.0027	102.1	

Formulas:

Standard TIC % Recovery = $((A-B)/((C/100)*D))*10^{-6}*100$

Standard TOC % Recovery = $((E-F)/((G/100)*H))*10^{-6}*100$

Sample TIC (ug C/ml or ug C/g) = (I-J)/(K*L/100)

Sample TOC (ug C/ml or ug C/g)= (M-N)/(O*P/100)

Matrix Spike Recoveries:

TIC % Recovery = (((Q-R)/(L/100))-S*T)*100/U

TOC % Recovery = (((Q-R)/(P/100))-S*T)*100/U

TC % Recovery = $((((Q^{TIC}-R^{TIC})/(L/100))-V^{TIC})+(((Q^{TOC}-R^{TOC})/(P/100))-V^{TOC}))*100/U^{TIC+TOC}$

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Pooled SD is the averaged SD for a recent list of 12 sample batches. MDL is based upon the Pooled SD. MDL = 3 x pooled SD.

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

TIC and TOC are measured; TC is the sum of the TIC and TOC results.

Sample Results

Note: Sample weights are on "as received" basis; i.e., wet weight

ACL Number	Client Sample ID	[I] Raw TIC (ug C)	[J] Blk (ug C)	[K] Sam wt (g)	TIC (ug C/g)	TIC RPD (%)	[M] Raw TOC (ug C)	[N] Blk (ug C)	[O] Sam wt (g)	TOC (ug C/g)	TOC RPD (%)	TC (ug C/g)	TC RPD (%)
01-01017	LS-16	2200	14	0.1201	18,533		765	54	0.1201	6,185		24,718	
01-01017 Dup	LS-16 Dup	2625	14	0.1499	17,735	4	1052	54	0.1499	6,955	12	24,690	0
01-01017 MS	LS-16 MS	2732	14	0.0939	see below		1468	54	0.0939	see below		see below	

(Note: For any TOC or TIC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit by [K])

Matrix Spike Results

ACL Number	Client Sample ID	[Q] Raw MS (ug C)	[R] MS Blk (ug C)	[S] Sam (ug C/g)	[T] MS Sam wt (g)	[V] Sample (ug C)	Spike wt (g)	[U] Spike (ug C)	MS % Recovery	
01-01017 MS	TIC Recovery	2732	14	18533	0.0939	1740	0.0082	983	**104.5**	TIC
	TOC Recovery	1468	54	6185	0.0939	581	0.0025	1000	**89.6**	TOC
	Total Carbon Recovery (TIC + TOC)							1983	**97.0**	TC

Reviewer/date: [signature] 10-02-01

HOT PERSULFATE WORKSHEET

Solid Client Hallen ASR 6107 Analyst MJ Steele Date 7/31/01

Procedure: PNL-ALO-381 Analyzer M&TE: WC01713 Balance M&TE: 360-06-01-023

alpha-D-Glucose
Aldrich lot# HY12603EY
solid
CMS # 53219 STd

Calcium Carbonate
JT Baker Lot N30628
solid
CMS# 161359 STd

alpha-D-Glucose
Sigma lot 58H01281
solid
CMS # 161713 MS/LCS

Calcium Carbonate
Mallinckrodt Lot# 4071 T34626
Solid
CMS# 161732 MS/LCS

Lab ID	Client ID	volume	sample wt (g)	TIC (ug)	TOC (ug)	comments
Blank			—	13	59	
↓			—	13	41	
Std			7.6 mg 1.4 mg	912	595	
↓			7.6 mg 1.4 mg	897	605	
01-1017	MS	8.2 mg 2.5 mg	0.0939	2732	1468	
	Sample		0.1201	2200	765	
	Duplicate		0.1499	2625	1052	
LCS		18.8 mg 2.7 mg	—	2255	1110	
Blk			—	15	61	
Std		6.9 mg 1.9 mg	—	834	754	

Carbon Bench sheet.xls

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client: Fiskum, Urie, Hallen
Project :
Work Pkg:
Analyzed:
ASR: 6014, 6031, 6107

MJ Steele 10/20/01

Analyzer M&TE: WA92040 -- 701
Balance M&TE: 360-06-01-023
TOC STD: VWR CMS#161356 0.10% Carbon <<[G]
TIC STD: VWR CMS# 161355 0.10% Carbon <<[C]

TIC	TOC	
12.5	52.7	<<< Blank Average (ug C)
2.7	14.3	<<< Blank Std Dev (ug C)
2.16	*5.8*	*<<< Pooled Std Dev (ug C)*
6.5	*17.3*	*<<< Method Det. Limit (ug C)*

Is Blank Std Dev < Method Det Limit?	
TIC	Yes
TOC	Yes

		Raw TIC (ug C)	Raw TOC (ug C)
Blanks:	Calibration blank (start of batch) 7-19-01	9.4	55.0
	Calibration blank (start of batch)7-19-01	9.8	50.0
	Calibration blank 7-19-01	14.0	35.0
	Calibration blank 7-19-01	13.0	35.0
	Calibration blank 7-20-01	15.0	61.0
	Calibration blank 7-20-01	10.0	58.0
	Calibration blank (end of batch) 7-20-01	16.0	75.0

		Total Inorganic Carbon (TIC)					Total Organic Carbon (TOC)				
		[A] Raw TIC (ug)	[B] Blk (ug)	[D] Std wt (g)	TIC % Rec		[E] Raw TOC (ug)	[F] Blk (ug)	[H] Std wt (g)	TOC % Rec	
Standards:	Calibration Standard (start of batch) 7-19-01	1007	12	1.0000	99.5		1007	53	1.0000	95.4	
	Calibration Standard (7-19-01)	970	12	1.0000	95.8		970	53	1.0000	91.7	
	Calibration Standard (7-19-01)	982	12	1.0000	97.0		995	53	1.0000	94.2	
	Calibration Standard (7-20-01)	1011	12	1.0000	99.9		1080	53	1.0000	102.7	
	Calibration Standard (end of batch) 7-20-01	970	12	1.0000	95.8		1081	53	1.0000	102.8	
				1.00000							
		[L] Average TIC % Rec >>>>			97.6	<<[L]	[P] Average TOC % Rec >>>>			97.4	<<[P]
QC	Blank Spike/LCS	1004	12	1.0000	101.6		1080	53	1.0000	105.5	
	Blank Spike/LCS	1015	12	1.0000	102.8		1050	53	1.0000	102.4	

Formulas:

Standard TIC % Recovery = $((A-B)/((C/100)*D))*10^{-6}*100$

Standard TOC % Recovery = $((E-F)/((G/100)*H))*10^{-6}*100$

Sample TIC (ug C/ml or ug C/g) = $(I-J)/(K*L/100)$

Sample TOC (ug C/ml or ug C/g)= $(M-N)/(O*P/100)$

Matrix Spike Recoveries:

TIC % Recovery = $(((Q-R)/(L/100))-S*T)*100/U$

TOC % Recovery = $(((Q-R)/(P/100))-S*T)*100/U$

TC % Recovery = $((((Q^{TIC}-R^{TIC})/(L/100))-V^{TIC})+(((Q^{TOC}-R^{TOC})/(P/100))-V^{TOC}))*100/U^{TIC+TOC}$

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.
The Pooled SD is the averaged SD for a recent list of 12 sample batches. MDL is based upon the Pooled SD. MDL = 3 x pooled SD.
If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".
TIC and TOC are measured; TC is the sum of the TIC and TOC results.

Sample Results

Note: Sample weights are on "as received" basis; i.e., wet weight

ACL Number	Client Sample ID (Liquids)	[I] Raw TIC (ug C)	[J] Blk (ug C)	[K] Sam Vol (ml)	TIC (ug C/ml)	TIC RPD (%)	[M] Raw TOC (ug C	[N] Blk (ug C)	[O] Sam Vol (ml)	TOC (ug C/ml)	TOC RPD (%)	TC (ug C/ml)	TC RPD (%)
01-0414	AW101 Stock	150	12	0.10	1,410		220	53	0.10	1,718		3,128	
01-0414 Dup	AW101 Stock	294	12	0.20	1,443	2	391	53	0.20	1,737	1	3,180	2
01-0414 Trip	AW101 Stock	1410	12	1.00	1,433		1800	53	1.00	1,794		3,227	
01-0414 MS	AW101 Stock	1719	12	0.50	see below		1899	53	0.50	see below		see below	
01-1203	AP1-Tc-0-C	570	12	0.10	5,715		217	53	0.10	1,687		7,402	
01-1203 Dup	AP1-Tc-0-C	600	12	0.10	6,023	5	207	53	0.10	1,584	6	7,607	3
01-1203 MS	AP1-Tc-0-C	1040	12	0.10	see below		675	53	0.10	see below		see below	
01-520 PB	Hot Cell Blank	22	12	1.00	10		41	53	1.00	-12 (<mdl)		10	
01-520 A	AP101-DFA	2620	12	0.50	5,346		855	53	0.50	1,648		6,993	
01-520 A Dup	AP101-DFA	2630	12	0.50	5,366	0	854	53	0.50	1,646	0	7,012	0
01-0520 B	AP101-DFB	2671	12	0.50	5,450		848	53	0.50	1,633		7,084	
01-0520 B Dup	AP101-DFB	2570	12	0.50	5,243	4	835	53	0.50	1,607	2	6,850	3
01-0520 B MS	AP101-DFB	3617	12	0.50	see below		1748	53	0.50	see below		see below	
01-01014	LS-12	4180	12	0.50	8,544		5885	53	0.50	11,977		20,521	
01-01014 Dup	LS-12	4185	12	0.50	8,554	0	5975	53	0.50	12,162	2	20,716	
01-01015	LS-13	1560	12	0.20	7,932		2290	53	0.20	11,486		19,418	
01-01015 Dup	LS-13	762	12	0.10	7,683	3	1160	53	0.10	11,370	1	19,053	2
01-01015 Trip	LS-13	1710	12	0.20	8,701		2415	53	0.20	12,128		20,829	
01-01016	LS-14	451	12	0.30	1,498		970	53	0.30	3,140		4,638	
01-01016 Dup	LS-14	746	12	0.50	1,504	0	1530	53	0.50	3,034	3	4,538	2
01-01016 MS	LS-14	1725	12	0.50	see below		2550	53	0.50	see below		see below	

(Note: For any TOC or TIC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit by [K])

Matrix Spike Results

ACL Number	Client Sample ID	[Q] Raw MS (ug C)	[R] MS Blk (ug C)	[S] Sam (ug C/ml)	[T] MS Sam Vol (ml)	[V] Sample (ug C)	Spike wt (g)	[U] Spike (ug C)	MS % Recovery	
01-0414 MS	TIC Recovery	1719	12	1410	0.50	705	1.0000	1000	104.4	TIC
	TOC Recovery	1899	53	1718	0.50	859	1.0000	1000	103.7	TOC
	Total Carbon Recovery (TIC + TOC)							2000	104.1	TC
01-1203 MS	TIC Recovery	1040	12	5715	0.10	572	0.5000	500	96.4	TIC
	TOC Recovery	675	53	1687	0.10	169	0.5000	500	94.1	TOC
	Total Carbon Recovery (TIC + TOC)							1000	95.2	TC
01-0520 B MS	TIC Recovery	3617	12	5450	0.50	2725	1.0000	1000	97.0	TIC
	TOC Recovery	1748	53	1633	0.50	817	1.0000	1000	92.4	TOC
	Total Carbon Recovery (TIC + TOC)							2000	94.7	TC
01-01016 MS	TIC Recovery	1725	12	1498	0.50	749	1.0000	1000	100.6	TIC
	TOC Recovery	2550	53	3140	0.50	1570	1.0000	1000	99.4	TOC
	Total Carbon Recovery (TIC + TOC)							2000	100.0	TC

Reviewer/date: MW [illegible] 10-02-01

HOT PERSULFATE WORKSHEET

Client Fiskum, S ASR 6014 Analyst MJ Steele Date 7-19-01

Procedure: PNL-ALO-381 Analyzer M&TE: WC01713 Balance M&TE: 360-06-01-023

TIC STD: Lot 0010016 CMS# 161355 TOC STD: Lot 0010285 CMS# 161356

	Lab ID	Client ID	volume	sample wt (g)	TIC (ug)	TOC (ug)	comments
		Blank 1			9.38	55	pipet check
		Blank 2			9.82	50	0.1ml # 111842
		Std 1	1ml 1ml	—	1007	1007	0.0995
	LCS	Std 2	1ml 1ml	—	1004	1080	0.0996
6014	01-0414	AW101 Stock	100 ul	0.1252	150	220	0.0998
	Dup ↓	↓	200 ul	0.2489	293.6	391	0.0997
	Trip ↓	↓	1ml	1.2388	1410	1800	
	01-0414 MS	1ml each	Spike .5ml	Sample .6224	1719	1899	1ml # 273514
							0.9912
6121	01-1203		100 ul	0.1254	570	217	0.9949
	Dup			0.1263	600	207	0.9954
	MS	.5ml each Spike	↓	0.1269	1040	675	0.9995
	01-0520	Hot Cell Blank	1ml	0.9910	22	41	0.5ml #23
	AP101-DF						0.5040
		STD	1ml each		970	970	0.5010
		Blank			14	35	0.5004
							0.4996
6031	01-0520 A	AP101-DFA	.5ml	.6241	2620	855	
	A Dup		.5ml	.6248	2630	854	
	01-0520 B	AP101-DFB	.5ml	.6348	2677	848	
	Dup	↓		.6279	2570	835	
	MS	1ml each Spike	↓	.6315	3617	~~2677~~ mjs 7-19-01	1748

Carbon Bench sheet.xls

HOT PERSULFATE WORKSHEET

Client ____________ ASR ____________ Analyst MJ Steele Date 7-19-01

Procedure: PNL-ALO-381 Analyzer M&TE: WC01713 Balance M&TE: 360-06-01-023

TIC STD: TOC STD:

Lab ID	Client ID	volume	sample wt (g)	TIC (ug)	TOC (ug)	comments
01-01014	LS-12	.5ml	0.6280	4180	5885	
↓	LS-12	.5ml	0.6313	4185	5975	
01-01015	LS-13	.2ml	0.2465	1560	2290	
↓	LS-13	.1ml	0.1226	762	1160	
	STd 1ml each			982	995	
	Blank			13	35	
7-20-01	Std 1	1ml each		1011	1080	
LCS.	Std 2	↓		1015	1050	
	Blank 1			15	61	
	Blank 2			10	58	
01-01015	LS-13	.2	0.2490	1710	2415	
01-01016	LS 14	.3	0.3157	451	970	
	Dup	.5	0.5313	746	1530	
↓	↓ MS	.5	0.5307	1725	2550	Spike 1ml each
	Std 3			970	1081	
	Blk 3			16	75	

Carbon Bench sheet.xls

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

PO Box 999, Richland, WA 99352

Client	R. Hallen	**Charge Code/Project:**	W57984 / 42365
RPL Numbers:	01-1014 to 01-1017	**ASR Number:**	6107
Analyst:	MJ Steele	**Analysis Date:**	8/22, & 8/23 2001

Procedure: PNL-ALO-380, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer"

M&TE: Carbon System (WD13071); Balance (360-06-01-023).

TOC/TIC/TC Results

RPL Number	Sample ID	TIC (a) MDL ugC/mL	TIC (b) Results ugC/mL	TOC MDL ugC/mL	TOC Results ugC/mL	TC MDL ugC/mL	TC Results ugC/mL
01-01014	LS-12	220	5,700	180	15,500	220	21,200
01-01014 Dup	LS-12					220	21,200
	RPD						0%
01-01015	LS-13	220	nd	180	22,300	220	19,000
01-01016	LS-14	90	850	180	3,550	90	4,400
01-01016 Dup	LS-14	90	900	180	3,370	90	4,270
	RPD				5%		3%
01-01015 MS	LS-13						107%
01-01016 MS	LS-14				105%		
BS/LCS	Blank Spike/LCS				96%		94%
RPL Number	**Sample ID**	**ugC/g**	**ugC/g**	**ugC/g**	**ugC/g**	**ugC/g**	**ugC/g**
01-01017	LS-16 Washed Solids	190	nd	400	22,700	190	28,000
01-01017 Dup	LS-16 Washed Solids	129	5,400	240	23,100	130	28,500
	RPD				2%	-	2%
01-01017MS	LS-16 Washed Solids				100%		111%
BS/LCS	Blank Spike/LCS				99%		104%

nd = not detected (i.e., TC results > TOC results)
(a) TIC MDL set to TC MDL
(b) TIC is determined by difference (TC - TOC)

The TOC/TIC analyses of the samples submitted under ASRs 6107 were to be performed by both the hot persulfate and furnace methods. This report presents the results from the furnace oxidation method and the results are compared to the results obtained from the hot persulfate method. Determination of total organic carbon (TOC) is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 750 °C for 30 minutes. The total carbon is determined on another aliquot of the sample by combusting at 1000 °C for 30 minutes. The total inorganic carbon is obtained by difference.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-380.

Q.C. Comments:

The calibration and QC standards for TC and TOC analysis are liquid or solid carbon standards or pure chemicals from JT Baker, Aldrich, Sigma, and Mallinckrodt. The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets.

The coulometer analysis system calibration is checked by analyzing calibration standards at the beginning, middle, and end of each day's run. The average recovery from these calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recovery for the two analysis days was 100%, and 98%.

System blanks were analyzed similarly to the calibration check, averaged, and subtracted from the sample 'raw data' results prior to calculating the final reported result. The TOC determination produced an average blank of 15 μgC. The TC determination produced an average blank of 54 μgC. The 54 μgC blank level is unusually high; however, the reproducibility of the blank was reasonably good (i.e., 49 to 59 μgC).

For each days analysis run, the QC for the analyses include sample duplicates, blank spikes (as a laboratory control sample), and matrix spikes.

Blank Spike/Laboratory Control Sample: The BS/LCS was within acceptance criteria of 80% to 120% required by the governing QA Plan for both the TC and TOC analysis of the liquid and solids samples.

Duplicates: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD), is within the acceptance criteria of the governing QA Plan (i.e., <20%).

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The TOC and TC matrix spike for both the liquids and solids samples demonstrates recoveries between 100% and 111%, which are within the acceptance criteria of 75% to 125%.

Furnace Results Compared to Hot Persulfate Results

RPL Number	Sample ID	TIC HP Results ugC/mL	TIC Furn Results[(a)] ugC/mL	TOC HP Results ugC/mL	TOC Furn Results ugC/mL	TC HP Results[(b)] ugC/mL	TC Furn Results ugC/mL
01-01014	LS-12	8,540	5,700	12,000	15,500	20,500	21,200
01-01015	LS-13	7,930	nd	11,500	22,300	19,400	19,000
01-01016	LS-14	1,500	850	3,140	3,550	4,640	4,400
RPL Number	**Sample ID**	**TIC HP Results ugC/g**	**TIC Furn Results[(a)] ugC/g**	**TOC HP Results ugC/g**	**TOC Furn Results ugC/g**	**TC HP Results[(b)] ugC/g**	**TC Furn Results ugC/g**
01-01017	LS-16 Washed Solids	18,500	130	6,190	22,700	24,700	28,000

nd = not detected (i.e., TOC result > TC result)

(a) TIC Furn is determined by difference (TC - TOC)

(b) TC HP is determined by sum (TIC + TOC)

The two method appear to produce comparable results for TC, with the furnace producing slightly higher results. However, there are significant differences between the TIC and TOC results reported by each method. The reason for the discrepancy between the hot persulfate method and furnace method is unknown, but it appears that the inorganic carbon, perhaps in the form of easily oxidized metal carbonates, is being combusted at 750 °C (as TOC) with the furnace method. Typically, the furnace method provides the best TC results and the hot persulfate the best TIC results, thus the TOC would be the difference between these measurements. Based on the furnace TC result for sample LS-16, the TOC result from the hot persulfate method may be about 30% low.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- For both the TC and TOC, the analysis Method Detection Limit (MDL) is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples. The standard deviation is multiplied by the Student's t values for n-1 degrees of freedom to establish the daily MDL. The sample MDL (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL.

Report Prepared by: [signature] **Date** 2-19-01

Review/Approval by: [signature] **Date** 1-3-02

Raw Data Calculation/Archive Information:
ASR 6019L&S 6025L 6031L 6107S.xls
ASR 6014 6104 5105 6106 6107 6155 6162 6192.xls

Battelle PNNL/RSE/Inorganic Analysis ... ICPAES Analysis Report

PO Box 999, Richland, Washington 99352

Project / WP#: 42365 / W57984
ASR#: 6107
Client: R. Hallen
Total Samples: 1 (solids)

RPL#:	01-1017	
Client ID:	"LS-16"	
Sample Preparation: PNL-ALO-115 (0.1g/100mL) PNL-ALO-129 (0.1g/28mL)		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 06-04-2001 (A0678)
07-03-2001 (A0693)

See **Chemical Measurement Center 98620 file:** ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

9-19-01
Reviewed by

Jerry Wagner 9-25-01
Concur

Sample LS-16 (washed solids) from Analytical Service Request (ASR) 6107 were prepared by KOH fusion per PNL-ALO-115 and by acid digestion per PNL-ALO-129. The fusion and digestion were performed in the Shielded Analytical Laboratory. The fusion used approximately 0.2 g of sample diluted to a final volume of 100 mL, and the acid digestion used approximately 0.1 g of sample diluted to a final volume of about 28 mL.

Table 2 in the ASR identified the analytes of interest, which include nearly all the standard analytes measured by the ICPAES. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those detected as part of the ICPAES analysis are reported, but have not been fully evaluated for QC performance. For those samples prepared from the KOH fusion process, neither K nor Ni (i.e., the fusion crucible material) is reported.

The attached ICPAES Results (4 pages) presents the final results. The fusion results are from measurement of the fusion preparations following an additional 2x dilution at the ICPAES. The acid digestion results are from measurements made at 1x or 2x dilution at the ICPAES, except for Mn and Sr which had to be diluted by at least 5x to ensure that the Mn and Sr were within the linear calibration range. The ICPAES measurement results are reported in µg/g of as-received solids and have been corrected for all dilutions resulting from sample processing. Two set of fusion results are included in the report. The initial results on the 06-04-2001 run produced very poor precision between the duplicates; the 07-03-2001 reanalysis of the fusion preparation confirmed that the poor precision was due to the sample heterogeneity or preparation error, and not due to the ICPAES analysis.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For the fusion processing, a fusion processing blank, laboratory control sample (Montana Soil SRM), and duplicate were prepared with the sample. No matrix spike was prepared since spiking of acid-based standards into the caustic fusion impacts the fusion preparation. Post spikes were used to assess matrix interferences. For the acid digestion processing, the QC included a matrix spike.

<u>Process Blank:</u>

Fusion: Concentrations of analytes of interest measured in the process blank were all within acceptance criteria of ≤ EQL (estimated quantitiation level) or less than ≤5% of the concentration in the sample.

Acid Digestion: No analytes of interest were detected in the acid digestion blank above the method detection limit (MDL), and clearly meets the processing blank acceptance criteria.

<u>Laboratory Control Sample (LCS):</u>

Fusion: The LCS for the fusion preparation is Montana Soil SRM 2710. This LCS does not contain all the analytes of interest, but is considered the best available material for evaluating the fusion processing. The LCS recoveries for analytes of interest were within the acceptance criteria of 80% to 120%, except Na which recovered slightly high at 122%.

Acid Digestion: The LCS for the acid digestion preparation is a blank spike prepared from two standard solutions (i.e., 010514i901 and 010514i902). This LCS include most, but not all of the analytes of interest defined by the ASR. The following analytes are not included in the LCS – B, Be, Ce, Co, Dy, Eu, Li, Nd, Sb, Sn, Th, V, and Y. For all other analytes of interest, the LCS recoveries were within the acceptance criteria, except Ag which recovered slightly low at about 73%.

Duplicate Relative Percent Difference (RPD):

Fusion: For those analytes of interest measured above the EQL, the RPDs were within the acceptance criteria of <20%, except Ca and Fe. Overall the fusion preparation demonstrated very poor precision between the duplicates. Since there was no results trend (i.e., all analytes in one sample being lower than the other, suggesting a weighing or processing error), sample heterogeneity and use of very small sample sizes is suspected to cause the poor precision.

Acid Digestion: For all analytes of interest measured above the MDL (and the EQL), the RPD were within the acceptance criteria.

Matrix Spiked Sample:

Fusion: No matrix spike is prepared in the fusion processing.

Acid Digestion: The matrix spike recoveries for the analytes of interest fall into three categories. 1) those analytes for which a matrix spike element was not included in the matrix spiking solution (see list under LCS), 2) those analytes that have spike concentrations less than 20% of the sample concentration (i.e., Al, Cr, Fe, Mn, Na, and Sr) making the spike recovery calculation meaningless, and 3) those analytes that demonstrated matrix spike recoveries within the acceptance criteria. For those analytes falling into categories 1) and 2), post spiking of the digestate (or in some cases serial dilution) is used to evaluate matrix interference issues.

Post-Spiked Samples (Spike A Elements):

Fusion: All post-spiked analytes of interest in samples tested were recovered within tolerance of 75% to 125%, except Al, Mn, and Sr (as well as K and Ni which are not reported for the fusion preparation). The post spike analysis uses a general spiking solution intended to be usable on the majority of sample analyzed by ICPAES. However, for the sample selected for post spiking, the spike concentration for Al, Mn, and Sr was less than 20% of the sample concentration and the recovery results are considered meaningless. For these analytes, the use of serial dilution results is required to evaluate potential matrix interferences.

Acid Digestion: All post-spiked analytes of interest in samples tested were recovered within tolerance of 75% to 125%, except Al, Mn, and Sr. The post spike analysis uses a general spiking solution intended to be usable on the majority of sample analyzed by ICPAES. However, for the sample selected for post spiking, the spike concentration for Al, Mn, and Sr was less than 20% of the sample concentration and the recovery results are considered meaningless. For these analytes, the use of serial dilution results is required to evaluate potential matrix interferences.

Project / WP#: 42365 / W57984
ASR#: 6107
Client: R. Hallen
Total Samples: 1 (solid)

	First	Last
RPL#:	01-01017	--
Client ID:	"LS-16"	--
Sample Preparation: PNL-ALO-115 & PNL-ALO-129 (SAL/vh)		

Revision 1 (added MS serial dilution data for Mn and Sr, added RPD values for Mn and Na for the fusion processing, updated BS and MS recovery values and serial dilution %D values, and editorial changes)

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 06-04-2001 (A0678)
07-03-2001 (A0693)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

B.M. Oliver 11/12/02
Preparer

MW Urie 11/13/02
Review and Concur

Sample LS-16 (washed solids) from Analytical Service Request (ASR) 6107 was prepared by KOH fusion per PNL-ALO-115 and by acid digestion per PNL-ALO-129. The fusion and digestion were performed in the Shielded Analytical Laboratory. The fusion used approximately 0.2 g of sample diluted to a final volume of 100 mL, and the acid digestion used approximately 0.1 g of sample diluted to a final volume of about 28 mL.

Table 2 in the ASR identified the analytes of interest (AOIs), which included nearly all of the standard analytes measured by the ICPAES instrument. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those specified as AOIs are reported, but have not been fully evaluated for QC performance. For those samples prepared from the KOH fusion process, neither K nor Ni (i.e., the fusion crucible material) are reported.

The attached ICPAES Results (5 pages) presents the final results. The fusion results are from measurement of the fusion preparations following an additional 2x dilution at the ICPAES. The acid digestion results are from measurements made at 1x or 2x dilution at the ICPAES, except for Mn and Sr which had to be diluted by at least 5x to ensure that the Mn and Sr were within the linear calibration range. The ICPAES measurement results are reported in µg/g of as-received solids and have been corrected for all dilutions resulting from the sample processing. Two set of fusion results are included in the report. The initial results from the 06-04-2001 run produced very poor precision between the duplicates; the 07-03-2001 reanalysis of the fusion preparation confirmed that the poor precision was due to sample heterogeneity or preparation error, and not due to errors in the ICPAES analysis.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For the fusion processing, a fusion processing blank, laboratory control sample (Montana Soil SRM-2710), and duplicate were prepared with the sample. No matrix spike was prepared since spiking of acid-based standards into the caustic fusion impacts the fusion preparation. Post spikes were used to assess matrix interferences. For the acid digestion processing, the QC included a matrix spike.

<u>Process Blank:</u>

Fusion: Concentrations of analytes of interest measured in the process blank were all within the acceptance criteria of <EQL (estimated quantitation level ≡ 10 x MDL) or less than <5% of the concentration in the sample.

Acid Digestion: No analytes of interest were detected in the acid digestion blank above the method detection limit (MDL).

<u>Laboratory Control Sample (LCS):</u>

Fusion: The LCS for the fusion preparation was Montana Soil SRM-2710. This LCS does not contain all of the analytes of interest, but is considered to be the best available material for evaluating the fusion processing. The LCS recoveries for all analytes of interest were within the acceptance criterion of 80% to 120%.

Acid Digestion: The LCS for the acid digestion preparation is a blank spike prepared from two standard solutions (i.e., 0105141901 and 0105141902). This LCS included most, but not all of the analytes of interest defined by the ASR. The following analytes are not included in the LCS; B, Be, Ce, Co, Dy, Eu, Li, Nd, Sb, Sn, Th, V, and Y. For all other analytes of interest, the LCS recoveries were within the acceptance criterion.

Duplicate Relative Percent Difference (RPD):

Fusion: For those analytes of interest measured above the EQL, the RPDs were within the acceptance criterion of <20%, except for Ba (on 6/4/01), Ca, and Fe. Overall the fusion preparation demonstrated very poor precision between the duplicates. Since there was no particular trend in the results (i.e., all analytes in one sample being lower than the other, suggesting a weighing or processing error), sample heterogeneity and use of very small sample size is suspected to be the cause of the poor precision.

Acid Digestion: For all analytes of interest measured above the MDL (and the EQL), the RPD were within the acceptance criterion.

Matrix Spiked Sample:

Fusion: No matrix spike is prepared in the fusion processing.

Acid Digestion: The matrix spike recoveries for the analytes of interest fell into three categories; 1) those analytes for which a matrix spike element was not included in the matrix spiking solution (see list under LCS), 2) those analytes that had spike concentrations less than 20% of the sample concentrations (i.e., Al, Cr, Fe, Mn, Na, and Sr) making the spike recovery calculation meaningless, and 3) those analytes that demonstrated matrix spike recoveries within the acceptance criterion of 75% to 125%. For those analytes falling into categories 1 and 2, post spiking of the digestate (or in some cases, serial dilution) must be used to evaluate matrix interference issues.

Post-Spiked Samples (Spike A Elements):

Fusion: All post-spiked analytes of interest in the samples tested were recovered within the acceptance criterion of 75% to 125%, except for Al, Mn, and Sr (as well as K and Ni which are not reported for the fusion preparation). The post spike analysis uses a general spiking solution intended to be usable on the majority of sample analyzed by ICPAES. However, for the present sample, the spike concentration for Al, Mn, and Sr was less than 20% of the sample concentration and thus the recovery results are considered meaningless. For these analytes, the use of serial dilution results is required to evaluate potential matrix interferences.

Acid Digestion: All post-spiked analytes of interest in samples tested were recovered within the acceptance criterion of 75% to 125%, except for Al, Mn, and Sr. As discussed above, the post spike analysis uses a general spiking solution intended to be usable on the majority of sample analyzed by ICPAES. However, for the present sample, the spike concentration for Al, Mn, and Sr was less than 20% of the sample concentration and thus the recovery results are considered meaningless. For these analytes, the use of serial dilution results is required to evaluate potential matrix interferences.

Post-Spiked Samples (Spike B Elements):

Fusion: All post-spiked analytes of interest in the sample were recovered within the acceptance criterion of 75% to 125%.

Acid Digestion: All post-spiked analytes of interest in the sample were recovered within the acceptance criterion of 75% to 125%.

Serial dilution (Percent Difference):

Fusion: Percent differences (%Ds) are listed for all analytes that had a concentration ≥ EQL in the diluted sample. Five-fold serial dilution was required for Al, Mn, and Sr since the post spike concentrations were less than 20% of the sample concentration (i.e., recoveries could not be evaluated). The %Ds were within the acceptance criterion of ±10% for all AOIs, including Al, Mn and Sr.

Acid Digestion: Percent differences (%Ds) are listed for all analytes that had a concentration ≥ EQL in the diluted sample. Five-fold serial dilution was required for Al, Mn, and Sr since the post spike concentrations were less than 20% of the sample concentration (i.e., recoveries could not be evaluated). The %Ds were determined from the 1x and 5x dilutions, and except for Mn and Sr, were within the acceptance criterion of ±10% for all AOIs. Unfortunately, %Ds could not be determined for Mn and Sr because these analytes exceeded the upper calibration range for the 1x dilution, and no additional serial dilution beyond 5x was performed. However, the matrix spike was analyzed at 1x, 5x, and 25x, and the %Ds calculated for Mn and Sr for the 5x and 25x dilutions were 3.2 and 3.4%, respectively. The quantity of spike added to the MS sample is significantly less than 20% of the sample concentration; therefore, the %Ds determined from the MS are considered to be representative of the LS-16 sample.

Other QC:

Measured concentrations for europium in the Initial Calibration Verification (ICV) runs were consistency high, at or just above the acceptance criterion of ±10%. This has minimal significance to the present data, however, since the concentration of europium in the sample was < MDL. All other instrument-related QC tests for the AOIs passed within the appropriate acceptance criteria.

Comments:

1) "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' by the IDL. Estimated quantitation limit (EQL) is equal to 10 x MLD.
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 μg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is two.

	Run Date=	7/3/2001	7/3/2001	7/3/2001	7/3/2001	7/3/2001
	Multiplier=	267.0	289.4	1447.0	507.2	1267.9
	RPL/LAB #=	01-1017-B	01-1017	01-1017 @5	01-1017-DUP @2	01-1017-DUP @5
Instr. Det. Limit (IDL)	Client ID=	*Process Blank*	*LS-16*		*LS-16-DUP*	
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	[15]		[13]	
0.060	Al	--	83,400		81,200	
0.050	B	--	[26]		--	
0.010	Ba	--	151		146	
0.010	Be	--	[10]		[9.7]	
0.250	Ca	--	3,210		3,390	
0.015	Cd	--	[32]		[33]	
0.200	Ce	--	[250]		[240]	
0.050	Co	--	--		--	
0.020	Cr	--	5,290		5,250	
0.025	Cu	--	[14]		--	
0.050	Dy	--	--		--	
0.100	Eu	--	--		--	
0.025	Fe	--	8,170		8,280	
2.000	K	--	--		--	
0.050	La	--	413		400	
0.030	Li	--	193		186	
0.100	Mg	--	--		[130]	
0.050	Mn	--	over range	68,200	over range	65,700
0.050	Mo	--	[53]		[55]	
0.150	Na	--	191,000		204,000	
0.100	Nd	--	[190]		735	
0.030	Ni	--	216		234	
0.100	P	--	1,600		1,770	
0.100	Pb	--	854		832	
0.750	Pd	--	[450]		[380]	
0.300	Rh	--	--		--	
1.100	Ru	--	--		--	
0.500	Sb	--	--		--	
0.500	Si	--	10,300		10,400	
0.500	Sn	--	--		--	
0.015	Sr	--	over range	94,900	over range	98,400
1.000	Th	--	--		--	
0.025	Ti	--	[36]		[39]	
2.000	U	--	[850]		--	
0.050	V	--	--		--	
0.050	Y	--	[70]		[66]	
0.050	Zn	--	309		299	
0.050	Zr	--	325		310	
Other Analytes						
0.250	As	--	--		--	
0.100	Bi	[29]	--		--	
0.250	Se	--	--		--	
0.500	Te	--	--		--	
0.500	Tl	--	--		--	
0.500	W	--	--		--	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible used for preparing samples.

	Run Date=	6/4/2001	6/4/2001	6/4/2001	7/3/2001	7/3/2001	7/3/2001
	Multiplier=	1058.8	1089.3	1029.9	1058.8	1089.3	1029.9
	RPL/LAB #=	01-01017-Blank-Ni @2	01-01017-Ni @2	01-01017-DUP-Ni @2	01-1017-Ni-BLK @2	01-1017-Ni @2	01-1017-Ni-DUP @2
Instr. Det. Limit (IDL)	Client ID=	*Process Blank-Ni*	*LS-16-Ni*	*LS-16-Ni-DUP*	*Process Blank-Ni*	*LS-16-Ni*	*LS-16-Ni-DUP*
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	[36]	[39]	--	[35]	[29]
0.060	Al	[240]	80,500	68,900	[210]	81,700	70,000
0.050	B	--	[67]	[66]	--	[61]	[58]
0.010	Ba	--	201	123	--	146	124
0.010	Be	--	--	--	--	--	--
0.250	Ca	--	3,680	2,780	[310]	3,410	2,770
0.015	Cd	--	[52]	[68]	--	[43]	[59]
0.200	Ce	--	[330]	[340]	--	[240]	[230]
0.050	Co	--	[56]	[120]	--	--	[100]
0.020	Cr	[37]	5,430	4,650	[35]	5,390	4,560
0.025	Cu	--	--	--	--	--	--
0.050	Dy	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--
0.025	Fe	335	8,970	7,310	327	8,990	7,280
2.000	K	na	na	na	na	na	na
0.050	La	--	[370]	[330]	--	[360]	[310]
0.030	Li	--	[190]	[170]	--	[180]	[160]
0.100	Mg	[110]	[220]	[220]	--	[180]	[160]
0.050	Mn	[94]	65,300	55,200	[95]	64,400	54,100
0.050	Mo	--	[82]	[86]	--	[71]	[72]
0.150	Na	3,040	186,000	205,000	3,560	191,000	213,000
0.100	Nd	[160]	[790]	[720]	[120]	[730]	[630]
0.030	Ni	na	na	na	na	na	na
0.100	P	[140]	2,170	2,210	[110]	2,070	2,070
0.100	Pb	[180]	[900]	[860]	[130]	[800]	[700]
0.750	Pd	--	--	[790]	--	--	--
0.300	Rh	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--
0.500	Si	--	15,300	13,400	--	15,500	13,500
0.500	Sn	--	--	--	--	--	--
0.015	Sr	--	89,800	75,400	--	95,200	79,000
1.000	Th	--	--	--	--	--	--
0.025	Ti	--	[100]	[130]	--	[97]	[130]
2.000	U	[2,500]	[2,800]	[3,300]	--	--	--
0.050	V	--	--	--	--	--	--
0.050	Y	--	[65]	[58]	--	[60]	[52]
0.050	Zn	--	[460]	[330]	--	[310]	[310]
0.050	Zr	--	[130]	[130]	--	[120]	[120]
Other Analytes							
0.250	As	--	--	--	--	--	--
0.100	Bi	--	--	[130]	--	--	[110]
0.250	Se	--	--	--	--	--	--
0.500	Te	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--
0.500	W	--	--	--	--	--	--

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible used for preparing samples.

QC Performance 6/4/01

Criteria>	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< ±10%
QC ID=	01-01017-Ni & 01-01017-Ni-D	LCS SRM-2710	MS (none)	01-01017-Ni + Post Spike A	01-01017-Ni + Post Spike B	01-01017-Ni @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Ag				99		
Al	15.7	99		nr		1.9
B				104		
Ba	48.1	93		98		
Be				**110**		
Ca	28.0	104		103		
Cd				104		
Ce					**103**	
Co				107		
Cr	15.4			117		3.1
Cu		88		81		
Dy					98	
Eu					104	
Fe	20.3	101		105		3.2
K	na	na		na		na
La					97	
Li				99		
Mg		107		109		
Mn	16.8	105		nr		4.1
Mo				104		
Na	9.9	96		112		4.3
Nd		nr			101	
Ni	na	na		na		na
P	2.0	101		107		
Pb		102		110		
Pd					**135**	
Rh					**116**	
Ru						
Sb				**110**		
Si	13.3	nr		111		
Sn					**124**	
Sr	17.4	98		nr		0.9
Th					**101**	
Ti		90		98		
U		nr			**99**	
V				100		
Y				98		
Zn		102		107		
Zr				102		
Other Analytes						
As				**114**		
Bi				100		
Se				**115**		
Te					**110**	
Tl				**105**		
W						

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible used for preparing samples.

QC Performance 7/3/01

Criteria>	<20%	<20%	80% - 120%	75%-125%	75%-125%	75%-125%
QC ID=	01-01017 & 01-01017-D	01-01017-Ni & 01-01017-Ni-D	BS	01-01017 & 01-01017-MS	01-01017 + Post Spike A	01-01017 + Post Spike B
Analytes	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec
Ag			81	104	102	
Al	2.4	15.5	101	nr	nr	
B					104	
Ba	3.8	15.9	101	97	101	
Be					**106**	
Ca	4.3	20.9	101	101	102	
Cd			101	100	101	
Ce						**112**
Co					106	
Cr	2.6	16.5	101	nr	103	
Cu			103	103	106	
Dy						103
Eu						110
Fe	3.6	21.0	104	nr	105	
K		na	92	93	99	
La	2.8		101	101		100
Li	2.5				103	
Mg			103	105	109	
Mn		17.3	104	nr	nr	
Mo					106	
Na	5.7	10.8	101	nr	101	
Nd	2.7		102	101		102
Ni	7.0	na	103	98	107	
P	1.7	0.1	100	101	101	
Pb	1.1		103	102	104	
Pd			99			**107**
Rh			101			**101**
Ru			**98**			
Sb					**101**	
Si	2.1	13.9	95	80	108	
Sn						
Sr		18.6	102	nr	nr	
Th						**103**
Ti			98	97	101	
U			101	104		**100**
V					**98**	
Y					104	
Zn	3.6		103	101	106	
Zr	2.3		101	102	102	
Other Analytes						
As					**105**	
Bi			100	100	102	
Se					**105**	
Te						
Tl					**105**	
W						

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible used for preparing samples.

(a) = Value for matrix spike at 5x/25x dilutions (see text).

QC Performance 7/3/01

Criteria>	<20%	75%-125%	75%-125%	< ±10%	< ±10%
QC ID=	01-01017 & 01-01017-D	01-01017-Ni + Post Spike A	01-01017-Ni + Post Spike B	01-01017 @1/@5 Serial Dil	01-01017-Ni @2/@10 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Diff	%Diff
Ag		97			
Al	2.4	nr		0.3	1.5
B		100			
Ba	3.8	95		0.5	
Be		**106**			
Ca	4.3	99			
Cd		99			
Ce			**104**		
Co		108			
Cr	2.6	93		4.0	4.6
Cu		78			
Dy			101		
Eu			107		
Fe	3.6	100		7.0	4.3
K		na			na
La	2.8		99		
Li	2.5	97			
Mg		105			
Mn		nr		3.2 (a)	5.2
Mo		101			
Na	5.7	91		0.6	2.2
Nd	2.7		103		
Ni	7.0	na			na
P	1.7	101		10.0	
Pb	1.1	104			
Pd			**124**		
Rh			**110**		
Ru					
Sb		**104**			
Si	2.1	105		0.7	
Sn					
Sr		nr		3.4 (a)	0.9
Th			**102**		
Ti		95			
U			**105**		
V		**97**			
Y		**94**			
Zn	3.6	102			
Zr	2.3	100			
Other Analytes					
As		**109**			
Bi		99			
Se		**110**			
Te					
Tl		**104**			
W					

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible used for preparing samples.

(a) = Value for matrix spike at 5x/25x dilutions (see text).

Serial Dilution:

Acid Digestion: Serial Dilution (@5x) was performed for Al, Mn, and Sr since the post spike concentrations for these analytes were <20% of the sample concentration (i.e., recoveries could not be evaluated). Aluminum demonstrated a percent difference (%D) within the acceptance criterion of ±10% comparing the 1x and 5x dilution results. Unfortunately, Mn and Sr exceeded the upper calibration range for the 1x dilution and no %D could be calculated, since no additional serial dilution (i.e., 25x) was analyzed for the 'LS-16 Sample'. However, the MS sample was analyzed at 1x, 5x, and 25x, and the %Ds calculated for Mn and Sr for the 5x and 25x dilutions were 3.2% and 3.4%, respectively. The quantity of spike added to the MS sample is significantly <20% of the sample concentration; therefore, the %Ds determined on the MS sample are considered to be representative of LS-16 Sample.

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Date: 6/20/01

Subject: Hydroxide Analyses for: R. Hallen
ASR: 6107

To: R. Hallen

From: L. Greenwood LRG

Samples of the filtrate composite and wash composite from the Sr/TRU Project AN-102/C-104 blend were analyzed for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed in duplicate using a Brinkman 636 Auto-Titrator. A 0.1186 N NaOH solution was prepared for use as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution. Three inflection points were observed for each sample generally corresponding to hydroxide, carbonate, and bicarbonate. The standard hydroxide recovery averaged 95% and a sample spike recovered at 96%. No hydroxide was detected in a reagent blank. The titration curves are included with the report.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

ASR # 6107

Hydroxide and Alkalinity Determination
Procedure: PNL-ALO-228 Equip # WB76843

Analyst: Lori P. Darnell 6-21-01

Reviewer: L R Greenwood 6-20-01

Summary Report

RPG #	Client ID		Concentration, moles First Point	Second Point	Third Point
01-1014	LS-12		0.21	1.29	0.93
01-1014	LS-12	Rep	0.19	1.28	0.96
		RPD	7%	1%	4%
01-1015	LS-13		0.21	1.26	0.87
01-1015	LS-13	Rep	0.18	1.28	0.89
		RPD	18%	1%	2%
01-1016	LS-14		0.057	0.24	0.18
01-1016	LS-14	Rep	0.053	0.22	0.12
		RPD	6%	6%	41%
Standard 1			95%		
Standard 2			95%		
MS-1015	Matrix spike		96%		
Blank			nd		

Note: Results are presented for the first, second, and third inflection points on the titration curves, as applicable. The first inflection point is generally associated with the hydroxide concentration. The second and third points generally represent the carbonate and bicarbonate concentrations.

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	R. Hallen	Charge Code/Project:	W57984/42365
ASR Number:	6107 Liquids	Sample Receipt Date:	05/11/2001
Sample Prep Date:	N/A	Sample Analysis Date:	06/13-14/2001
Analyst:	MJ Steele		

Preparation Procedure: N/A
Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.\\

The final ion chromatography results ASR 6107 Liquid Samples (01-01014 through 01-01016) are presented in Table 1. Table 1 includes the samples, duplicates, processing blank (dilution liquid), low level standard, and laboratory control standard results.

No footnote for formate/acetate!

Table 1: Anion Analysis Results – ASR 6107 Liquids

RPL Number	Sample ID	F µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL	0.13	0.13	0.25	0.13	0.25	0.25	0.25	0.25
01-01014 Dilution Blank		< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	< 0.25	< 0.25
	EQL	625	125	625	125	1250	250	250	250
01-01014	LS-12	4,300	1,960	43,600	< 125	106,000	3,260	6,710	1,530
01-01014 DUP	LS-12 Dup	4,500	1,920	43,900	< 125	107,000	3,280	6,710	1,530
	RPD	4%	2%	1%	n/a	1%	1%	0%	0%
01-01015	LS-13	4,400	1,880	41,200	< 125	99,500	3,620	6,430	1,460
01-01015 MS %Rec	LS-13 MS %Rec	114%	104%	106%	106%	110%	97%	100%	107%
	EQL	125	25	250	25	250	250	50	50
01-01016	LS-14	4,800	380	7,290	< 25	16,900	340	1,060	2,770
01-01016 MS %Rec	LS-14 MS %Rec	111%	104%	108%	107%	110%	100%	101%	109%
LLS %Rec		106%	111%	102%	101%	98%	98%	98%	108%
LCS %Rec		97%	100%	97%	98%	93%	91%	91%	100%
Fluoride exhibits significant interference from unknown anions making quantitation difficult. Fluoride results are maximum value.									
EQL = Estimate quantitation limt; based on lowest calibration standard times all dilution factors used to calculate the reported results. No results below the EQL are reported.									

The sample was prepared for ion chromatography anion analysis by dilution at 200-fold to 5000-fold in order to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the sample as dilutions less than 200-fold. The estimated quantitation limits which are based on the lowest calibration standard and the dilutions used for reporting the results are provided in Table 1.

Q.C. Comments:

Duplicates: No duplicate was provided. However, one sample was split and analyzed in duplicate. The duplicate relative percent difference (RPD) meets the acceptance criteria of <20%.

RT HALLEN
Date 8/22/01
Route
File 41-073
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Matrix Spike (HCV 010328): A matrix spike was prepared from two samples all anion recoveries were within the 75% to 125% recovery acceptance criteria, as shown in Table 1.

Laboratory Control Sample-LSC/BS (HCV 010328 @4x): A Blank Spike (i.e., the spike solution used to prepare the matrix spike samples) was prepared and measured at the same time as the Matrix Spike sample and demonstrated recoveries within the 90% to 110% acceptance criteria.

Low Level Standard (LLS/LCV 010328): As shown in Table 1, the LLS meets the acceptance criteria of 75% to 125% recovery.

System Blank/Processing Blanks: Ten system blanks were processed during the analysis of the liquid sample. No anions were detected in the system blanks above the estimate quantitation level.

Quality Control Calibration Verification Check Standards (ICV 010328): Ten mid-range verification standards were analyzed throughout the analysis runs. Except for four oxalate measurements, all anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. The oxalate measurements that failed produced a recovery from 111% to 112%.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Report Prepared by: M W Urie Date 7-18-01

Review/Approval: MJ Steele Date 7-19-01

Archive Information:

Files: ASR 6107 Hallen.doc	ASR 6104, 6106, 6107 REP.xls

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	R. Hallen		Charge Code/Project:	W57984/42365
ASR Number:	6107 Solids		Sample Receipt Date:	05/09/2001
Sample Prep Date:	N/A		Sample Analysis Date:	06/20/2001
Analyst:	MJ Steele			

Preparation Procedure: PNL-ALO-103, "Water Leach of Sludges, Soils, and other Solids Samples"
Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

The final ion chromatography results ASR 6107 Solid Sample (01-01017) is presented in Table 1. Table 1 includes the samples, duplicates, matrix spikes,hot cell processing blank, low level standard, and laboratory control standard results.

Table 1: Anion Analysis Results – ASR 6107 Solid

RPL Number	Sample ID	F (1) µg/g	Cl µg/g	NO_2 µg/g	Br µg/g	NO_3 µg/g	PO_4 µg/g	SO_4 µg/g	C_2O_4 µg/g
	EQL	12	12	24	12	24	24	24	24
01-01017 PB	Hot Cell Blank (2)	< 12	< 12	< 24	< 12	< 24	< 24	< 24	< 24
	EQL	2,100	210	430	210	430	430	430	430
01-01017	LS-16	37,700	1,180	10,500	< 210	26,200	1,660	2,880	36,200
01-01017 Dup	LS-16 Dup	38,200	1,190	11,200	<210	27,900	2,420	2,900	36,100
	RPD	1%	1%	7%	n/a	6%	37%	0%	0%
01-01017 MS %Rec	LS-16 MS %Rec (3)	n/a	94%	n/a	97%	n/a	N.R.	122%	n/a
01-01017 PS 1 (1200x) %Rec	LS-16 PS1	106%	105%	105%	105%	101%	99%	99%	106%
01-01017 PS 2 (400x) %Rec	LS-16 PS2	113%	104%	104%	105%	102%	100%	100%	108%
LLS %Rec		104%	104%	101%	100%	97%	98%	97%	106%
LCS/BS (Hot Cell) %Rec		90%	108%	94%	91%	91%	90%	92%	97%
LCS (Lab) %Rec		103%	102%	101%	102%	97%	97%	96%	105%

(1) The fluoride results should be considered the upper bound concentration for the fluoride. Significant peak distortion of the fluoride peak suggests the presence of co-eluting anion(s), possibly formate or acetate.

(2) The hot cell blank reported in ug/g based on average hot cell sample dilution/leaching factor.

(3) MS results are considered qualitative. MS concentrations at or below the EQL following hot cell and IC dilutions.

n/a = MS level < 20% of sample concentration or RPD not calculated since sample and/or duplicate <EQL.

N.R. = No MS concentrations detected above sample concentration, suspected matrix interference.

EQL = Estimate quantitation limt; based on lowest calibration standard times all dilution factors used to calculate the reported results. No results below the EQL are reported.

RPD = Relative percent difference

The sample was prepared for ion chromatography anion analysis by leaching at approximately a 10:1 ratio with distilled de-ionized water. Once the leachates were prepared they had to be diluted an additional 20-fold to 200-fold in order to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the sample at dilutions less than 20-fold. The estimated quantitation limits (EQL) which are based on the lowest calibration standard and the dilutions used for reporting the results are provided in the Table 1.

Date 8/22/01
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R. J. Hallen

Q.C. Comments:

Duplicates: A sample and duplicate leach were performed in the hot cell and these duplicates analyzed. The duplicate relative percent difference (RPD) meets the acceptance criteria of <20% for all analytes measure above the EQL..

Matrix Spike (HCV 010328): A matrix spike (MS) was prepared in the hot cell. Since the MS required an additional 20-fold dilution at the IC workstation, the MS concentration was at or near the EQL. The MS concentration was less than 20% of the sample concentration for fluoride, nitrite, nitrate, and oxalate, and no MS recovery was calculated. Even at the low MS concentrations, chloride, bromide, and sulfate recoveries were within the 75% to 125% recovery acceptance criteria. The phosphate MS was unable to be recovered, suggesting some type of matrix interference or precipitation on leaching. Since the MS concentrations following dilution were so low, post spikes (PS) were prepared from the same spiking solutions and analyzed. The PSs demonstrated recoveries within the 75% to 125% acceptance criteria.

Laboratory Control Sample-LCS/BS (HCV 010328 @4x): Both the hot cell LCS/BS and the laboratory LCS/BS were prepared and measured at the same time as the MS sample. Both LCSs demonstrated recoveries within the 90% to 110% acceptance criteria.

Low Level Standard (LLS/LCV 010328): As shown in Table 1, the LLS meets the acceptance criteria of 75% to 125% recovery.

System Blank/Processing Blanks: Five system blanks were processed during the analysis of the solid sample. No anions were detected in the system blanks above the estimate quantitation level.

Quality Control Calibration Verification Check Standards (ICV 010328): Five mid-range verification standards were analyzed throughout the analysis runs. All anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Report Prepared by: MW Urie Date 7-20-01

Review/Approval: MJ Steele Date 7-20-01

Archive Information:

Files: ASR 6107 Hallen solids.doc	ASR 6107 Solids RR (010620).xls

Battelle, PNNL / RPG / Inorganic Analysis ... Mercury Data Report

Project / WP#:	42365 / K88408
ASR#:	6107
Client:	Rich Hallen
Total Samples:	1

RPL #	Client ID
01-01017	LS-16

Procedure: RPG-CMC-131 Rev. 0, *Mercury Digestion*
RPG-CMC-201 Rev. 0, *Mercury Analysis*

M&TE Number:	WD30853	CETAC, Mercury Analyzer, Model M-6000A
	360-06-01-029	Mettler AT400 Balance

Digestion Date: 8/1/01
Analysis Date: 8/1/01

Analysis File: 01080101.DB

Analyst: LMP Thomas

For Calibration and Maintenance Records, see Chemical Measurement Center 98620 RIDS

MW Thomas 9-7-01
Reviewed By / Date

One sample, LS-16, was submitted for mercury analysis. The sample was aliquoted in the hot cell, prepared by digestion in a fume hood, and analyzed by cold vapor atomic absorption spectroscopy (CVAA). The analysis of this sample included one duplicate and matrix spike. Quality control check standards relative to the sample preparation and instrument performance were also analyzed.

1. Analysis

Results from the analysis of the LS-16 sample, are provided in the table below. The concentration is reported in μg of mercury per gram of wet solids.

RPD success criteria: < 20%				
RPL #	Sample ID	Det Limit μg/g	Measured μg/g	RPD %
01-01017	LS-16	0.010	2.33	
01-01017	LS-16 DUP	0.015	2.81	19
Average			2.57	
Preparation DF (mLs/g)			157	
Analysis DF (v/v)			10	
DF - dilution factor				

2. Quality Control

Duplicate (DUP). The RPD between the LS-16 samples meets the success criteria.

Matrix Spike (MS). The matrix spike result does not meet the success criteria. This may be due to the concentration of the matrix spike, which is very small relative to the concentration of the sample.

MS success criteria: 75% to 125% of expected value					
RPL ID	Sample ID	Spike μg/g	Sample μg/g	Measured μg/g	Recovery %
01-01017	LS-16 MS	0.26	2.57	3.74	459

Preparation Blank (PB) and Laboratory Control Standard (LCS). The results of the PB and LCS analysis are presented in the table below. A PB, typically generated during the aliquoting of the samples in the hot-cell, was not prepared. The PBs from two other sample batches are reported here. These PBs were generated at a different time, but were digested with the LS-16 samples. The results of the PBs are based on the average sample size. The PBs and LCS meet the success criteria.

PB success criteria: < EQL		LCS success criteria: 90% - 110% of expected value		
Success Criteria μg/g	Measured μg/g	Expected μg/g	Measured μg/g	Recovery %
<0.079	< 0.012	1.40	1.35	96
<0.079	< 0.012			

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards. The ICB/CCB standards meet the success criteria.

ICB/CCB success criteria: < EQL		
Sample ID	**Criteria µg/L (1)**	**Measured µg/L (1)**
ICB	<0.05	<0.05
CCB 1	<0.05	<0.05
CCB 2	<0.05	<0.05
CCB 3	<0.05	<0.05
CCB 4	<0.05	<0.05
(1) Units are based on per liter of sample at the instrument.		

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards. The ICV/CCV standards meet the success criteria.

ICV/CCV success criteria: 90% to 110% recovery Calibration Range: 0 - 5 µg/L		
Sample ID	**Measured µg/L(1)**	**Recovery %**
Expected	2.00	
ICV	2.01	101
CCV 1	2.07	104
CCV 2	2.07	103
CCV 3	2.07	104
CCV 4	2.09	104
(1) Units are based on per liter of sample at the instrument.		

Low-Level Standard (LLS). The LLS meets the success criteria.

LLS success criteria: 75% to 125% recovery Lowest calibration std: 0.05 µg/L		
Expected µg/L(1)	**Measured µg/L(1)**	**Recovery %**
0.050	0.039	78
(1) Units are based on per liter of sample at the instrument.		

3. Comments

a). The mercury results have been corrected for all dilution factors performed on the sample during preparation and analysis.

b). The detection limit is based on the manufacturer's recommendations for non-complex, aqueous matrices. The estimated quantitation limit (EQL) is defined as the lowest calibration standard. Actual detection limits or quantitation limits for specific matrices may be determined, if requested.

c). Routine precision and bias is typically ±15% or better for non-complex aqueous samples that are free of interference.

Table S.1. (contd)

Analyte	Measure Method	Supernatant Average µg/mL	Supernatant Data Flag (a)	WCS Average µg/g	WCS Data Flag (a)	UDS Average µg/g WCS	UDS Average µg/g DS
W	ICP-MS	n/m		4.23e+01	X	4.2e+01	2.3e+02
Y	ICP-AES	3e+00	U	2.60e+01	J	2.6e+01	1.4e+02
Y	ICP-MS	n/m		2.58e+01		2.6e+01	1.4e+02
Zn	ICP-AES	3.30e+00	J	7.90e+01		7.7e+01	4.2e+02
Zr	ICP-AES	3e+00	U	7.80e+01	X	7.8e+01	4.3e+02

WCS = wet centrifuged solids
DS = dry centrifuged solids
UDS = undissolved solids
n/m = not measured
EDTA=ethylenediaminetetraacetic acid
HEDTA= N-(2-hydroxyethyl)ethylenediaminetriacetic acid
ED3A=ethylenediaminetriacetic acid
NTA=nitrilotriacetic acid
IDA=iminodiacetic acid;
D2EHP= bis-(2-ethylhexyl) phosphate.

Shaded areas represent analytes measured that were not listed in the TS (i.e., opportunistic analytes)

(a) Data flags: U = undetected; J = estimated value; B = analyte in blank above the blank acceptance criteria; X = QC deficiency
(b) Not calculated. Analysis of WCS for analyte no specified in TS.
(c) Not calculated. TS required analysis of analyte in WCS; however insufficient WCS available for analysis. BNI prioritized analyses to be performed on limited WCS material.
(d) Fluoride results should be considered the upper bound concentration for the fluoride. Significant peak distortion of the fluoride peak suggests the presence of co-eluting anion(s), possibly formate or acetate.
(e) For TOC and TIC: P=by hot persulfate method
F=by furnace method/TIC by difference (TC – TOC).
(f) Uranium measured in the supernatant and centrifuged solids by KPA and ICP-MS, respectively.
(g) The total Cs concentration is estimated in the solids based on the assumption the Cs isotopic distribution in the solids is equivalent to the isotopic distribution in the supernatant. Concentration is thus based on the relative contribution of 137Cs in the solids.
(h) Oxalate determined by inorganic IC method.
(i) The calculated UDS results <0; the UDS results set to <MDL of the WCS.

Pacific Northwest National Laboratory (PNNL) // Battelle Northwest
Radiochemical Science and Engineering (RSE)

Inorganic Analysis - Mercury Data Report

Project / WP#:	42365 / K88408
ASR#:	6107
Client:	Rich Hallen
Total Samples:	1

RPL #	Client ID
01-01017	LS-16

REVISION 1: Clarification of PB preparation and analysis

Procedure: RPG-CMC-131 Rev. 0, *Mercury Digestion*
RPG-CMC-201 Rev. 0, *Mercury Analysis*

M&TE Number: WD30853 CETAC, Mercury Analyzer, Model M-6000A
360-06-01-029 Mettler AT400 Balance

Digestion Date: 8/1/01
Analysis Date: 8/1/01

Analysis File: 01080101.DB

Analyst: LMP Thomas

Supporting data and records are located in the data package stored in the project file. Balance calibration records can be found in the Radiochemical Science and Engineering RIDS.

LMP Thomas — 11-11-02
Data Report Prepared By — Date

MW Urie — 11-13-02
Reviewed By — Date

One sample, LS-16, was submitted for mercury analysis. The sample was sub-sampled in the hot cell, prepared by digestion in a fume hood, and analyzed by cold vapor atomic absorption spectroscopy (CVAA). The analysis of this sample included one duplicate and matrix spike. Quality control check standards relative to the sample preparation and instrument performance were also analyzed.

1. Analysis

Results from the analysis of the LS-16 sample, are provided in the table below. The concentration is reported in µg of mercury per g of dry solid.

RPD success criteria: < 20%				
RPL #	Sample ID	Det Limit µg/g	Measured µg/g	RPD %
01-01017	LS-16	0.023	5.31	
01-01017	LS-16 DUP	0.029	5.46	2.7
	Average		5.39	
	Preparation DF (mLs/g)		333	
	Analysis DF (v/v)		10	
DF - dilution factor				

2. Quality Control

Duplicate (DUP). The RPD between the LS-16 samples meets the success criteria.

Matrix Spike (MS). The matrix spike result does not meet the success criteria. This may be due to the concentration of the matrix spike, which is very small relative to the concentration of the sample.

MS success criteria: 75% to 125% of expected value					
RPL ID	Sample ID	Spike µg/g	Sample µg/g	Measured µg/g	Recovery %
01-01017	LS-16 MS	0.54	5.39	7.87	460

Preparation Blank (PB) and Laboratory Control Standard (LCS). The results of the PB and LCS analysis are presented in the table below. Two PBs were prepared and analyzed with this samples set. The results of the PBs are based on the average sample size. The LCS is NIST SRM ~~2709~~ 2790 11/14/02, San Joaquin Soil. The PBs and LCS meet the success criteria.

PB success criteria: < EQL		LCS success criteria: 90% - 110% of expected value		
Success Criteria µg/g	Measured µg/g	Expected µg/g	Measured µg/g	Recovery %
<0.17	< 0.026	1.40	1.35	96
<0.17	< 0.026			

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards. The ICB/CCB standards meet the success criteria.

ICB/CCB success criteria: < EQL		
Sample ID	Criteria µg/L [1]	Measured µg/L [1]
ICB	<0.05	<0.05
CCB 1	<0.05	<0.05
CCB 2	<0.05	<0.05
CCB 3	<0.05	<0.05
CCB 4	<0.05	<0.05
[1] Units are based on per liter of sample at the instrument.		

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards. The ICV/CCV standards meet the success criteria.

ICV/CCV success criteria: 90% to 110% recovery Calibration Range: 0 - 5 μg/L		
Sample ID	**Measured μg/L(1)**	**Recovery %**
Expected	2.00	
ICV	2.01	101
CCV 1	2.07	104
CCV 2	2.07	103
CCV 3	2.07	104
CCV 4	2.09	104
(1) Units are based on per liter of sample at the instrument.		

Low-Level Standard (LLS). The LLS meets the success criteria.

LLS success criteria: 75% to 125% recovery Lowest calibration std: 0.05 μg/L		
Expected μg/L(1)	**Measured μg/L(1)**	**Recovery %**
0.050	0.039	78
(1) Units are based on per liter of sample at the instrument.		

3. Comments

a). The mercury results have been corrected for all dilution factors performed on the sample during preparation and analysis.

b). The detection limit is based on the manufacturer's recommendations for non-complex, aqueous matrices. The estimated quantitation limit (EQL) is defined as the lowest calibration standard. Actual detection limits or quantitation limits for specific matrices may be determined, if requested.

c). Routine precision and bias is typically ±15% or better for non-complex aqueous samples that are free of interference.

Battelle

. . . *Putting Technology To Work*

Date: 09/12/01

		Client ID	Sample RPG ID		
Subject:	Se-79 Analysis Report on:	LS-16	01-1017	ASR	6107
				WP#	W57984
Project:	Sr/TRU				

To: Rich Halen

Se-79 was measured according to procedure PNL-ALO-440 and the results are listed below. Since Se-79 is not available as a radioactive standard, Se carrier was used in the analysis for establishing the yield and C-14 was used to establish the instrument efficiency since it has a very similar beta energy. The samples were solubilized in the hot cells with a KOH-KNO3 fusion according to procedure PNL-ALO-115. A 1 mL aliquot of the fusion solution provided to the lab was analyzed. The gravimetric recoveries for the lab blank, hot cell blank, sample, and duplicate, are listed below. The Se-79 activities were measured by liquid scintillation counting according to procedure PNL-ALO-474. The beta energy spectra did not show any definite peaks for Se-79; however, the count rates were elevated in the Se-79 energy window and there was no evidence for any other beta contamination. The process blank activity exceeded both the sample and duplicate counting rates. The MDC value exceeded the sample activities for the lab blank and sample duplicate; hence, the MDC values should be reported for these samples. Since Se-79 is not available as a standard, it was not possible to provide LCS or matrix spikes.

		Se	Se-79 Result	Se-79 1s TPU	Se-79 MDC	
I.D.	Client ID	Recovery	uCi/g	uCi/g	uCi/g	TPU,%
Reag BlK		0.83	1.41E-4	1.99E-4	6.55E-4	141%
01-1017-PB		0.77	1.85E-3	2.50E-4	7.06E-4	14%
01-1017	LS-16	0.43	1.83E-3	4.19E-4	1.27E-3	23%
01-1017 -dup	LS-16 dup	0.68	4.97E-4	2.41E-4	7.69E-4	48%

Prepared by: [signature] Date: 9-12-01

Reviewed by: [signature] Date: 9-13-01

Se-79 Analysis --- via RPG-CMC-440   ASR # 6107

This procedure involves an anion/cation exchange to remove most radiochemical interferences followed by a Selenium Bromide distillation and minor interferences elimination by the reduction of Se to elemental form. The ppt. recovered is used for gravimetric yield correction. The Se is finally dissolved and counted by liquid scintillation using C-14 (prep'd in the same matrix configuration) for calibration since its beta energy max. of 156 keV is very similiar to Se-79 at 149 keV.

Se-79 Procedure Flow Diagram

Add 20 mg elemental Se (2 mL of 10mgSe/mL to each aliquot for analysis

.Prep plastic disposable columns: 1mL AG--50W: X 8 100-200 mesh H+ + 1ml AG-1, X-4 100-200 Cl-

Pass sample through column and collect in 35 mL vial. Wash with 4-1ml portions of 0.5 M HNO3

Add 10ml hydroxlamaine hydrochloride to sample in a glass vial. Cap and place the vial in a heating block and heat @ 90C for ~30 min (I.e. just until black ppt forms).

.Dissove ppt in a few drops of conc HNO3. Add 5 mL of water and repeat the NH2OH.HCl addition and precipitation. Then dissolve in ~ 0.5 M HNO3 for transfer to distillation flask.

Set up still by placing Nitrogen purge ~100ml boiling flask in heating mantle connected to a variable power supply and supported on a ring stand.

Transfer the solution to the boiling flask rinsing the 35mL vial with 2-5mL portions of concentrated HBr.

Place a rinsed (clean) 35ml vial on the exit port of the boiling flask. Add 5ml of 25% hydroxlamaine hydrochloride solution to this vial. Cool this vial in an ice bath.

Connect nitrogen supply to top of flask and set flow to about 5 bubbles/sec. Then turn on power supply and adjust to 110 V amd distill for at least 35 min.

Add 5 ml additional hydroxlamaine hydrochloride solution to the collection vial. Cap and place the vial in a heating block and heat for a couple hours. Black ppt. forms

Prep. and tare- weigh filter disks Centrifuge, decant most of solution and transfer black ppt. using ethanol to the top of filter mounted on vaccum system.

Dry ppt. and obtain final wt. for Se grav. recovery. 20 mg =100% .

Transfer filter + ppt. To glass LCS vial, add 2-3 drops of conc HNO3, allow to dissolve completely, then take to dryness. Add 2 mL 0.1 M HCL and 10 ml of LSC cocktail, place on labeled caps.

Prepare C-14 reference stds. similarly and count all on Packard LSC 2550 for 2 X 30 min. (Prog. # 6).

Analysis raw data:

Sample ID.	Initial Sam. Vol. (mL)	Diluted to	Tare Wt (mg) Filter + holder	Final Wt. (mg)	Net Se Wt.	Recovery %		
Reag BlK	1.00		747.6	764.1	16.5	83%		
01-1017-PB	1.00	Process Blk	751.9	767.2	15.3	77%		
01-1017	1.00		759.6	768.3	8.7	43%		
01-1017 -dup	1.00		775.9	789.5	13.6	68%		
01-1613	1.00		747.4	763.9	16.5	83%		
01-1842	1.00		752.7	760.2	7.5	38%		
01-1843	1.00		751.3	763.9	12.6	63%		
01-1843-dup	1.00		760.3	773.8	13.5	68%		

Pipet verify check	0.1 mL	1.0 mL
Pipet #	78868	33
	0.0996	1.022
	0.0985	1.0227
	0.1014	1.017
Ave	0.0998	1.0206
Std Dev.	0.0015	0.0031

[signature] 9/12/01
Entered by   Date

[signature] 9/12/01
Reviewed by   Date

Se-79 Analysis

WP# W57984

ASR # 6107 Prep Type washed solids Data File: r:\radchem\se79\se-6107.xls

Analysis for Se-79 was performed using PNL-ALO-440. This procedure involves an anion/cation exchange to remove of most radiochemical interferences followed by a Selenium Bromide distillation and minor interferences completely eliminated in the reduction of Se to elemental form. The ppt. recovered is used for gravimetric yield correction. No vendor supplied Se-79 source material is available, therefore C-14 was chosen for calibration since its beta energy max. of 156 keV is very similiar to Se-79 at 149 keV.
W-115-1, a secondary dilution of NIST C-14 SRM 4222, was used for the efficiency calibration of the liquid scintillation counter. These calibration standards were prepared in the same geometry as the prepared samples and at the same time the batch was prepared to monitor efficiency of the cocktail over time. Volume of W-115-1 used was 0.1ml.

Process Data: Start Date: 9/4/01
Se Carrier: Selenium Std. @ 10,000 ppm
CMS # 126666 Inor. Ventures Std
Vol. added: Expires: 1-Mar-01
2 mL = 20.0 mg

Performance checks
Balance # 37060155
Pipet # 78868
Lab Loc. 525

Sample ID	Leached Sample Vol (mL)		Total dil. volume (mL)	Diln. Aliquot (mL)	Tare Wt. of filter & holder (mg)	Gross Wt. of filter plu Se ppt.(mg)	Net Wt. of Se (mg)	Wt. of Se added (mg)	Grav. Recovery %
Reag BlK	1.000				747.6	764.1	16.5	20.00	82.5
01-1017-PB	1.000				751.9	767.2	15.3	20.00	76.5
01-1017	1.000				759.6	768.3	8.7	20.00	43.5
01-1017 -dup	1.000				775.9	789.5	13.6	20.00	68.0
01-1613	1.000				747.4	763.9	16.5	20.00	82.5
01-1842	1.000				752.7	760.2	7.5	20.00	37.5
01-1843	1.000				751.3	763.9	12.6	20.00	63.0
01-1843-dup	1.000				760.3	773.8	13.5	20.00	67.5

RG Brown 9/12/01
Entered by Date

LRGreenwood 9/12/01
Reviewed by Date

SELENIUM-79 CALCULATIONS FOR LIQUID SAMPLES

Procedure PNL-ALO-440

Data file name:	r:\radchem\se79\se-6107.xls	Entered by: rgs	
Date Counted	09/11/01	Date Calc'd	09/12/01
Sample Counting Time,min.	100.00		

C-14 is used for efficiency since Beta Emax is same as Se-79. T1/2 = 5715. yr

C-14 Std No.	W-115-1	Activity=	266700	dpm/ml
Reference date:	12/01/90	error=	400	dpm/ml

I.D.	aliquot ml	error ml	Se-79 R.O.I. cpm	1S%
Reag. Blank #1	1.000	0.0000	11.45	2.96
Blank 2	1.000	0.0000	12.15	2.87
		Avg =	***11.80***	cpm
			0.34	cpm

				1S%	efficiency	efficiency error
100ul Std - C-14 Spk 1	0.100	0.0003	19077.1	0.07	***0.72***	***0.0025***
100ul Std -C-14 Spk 3	0.100	0.0003	19306.0	0.07	***0.72***	***0.0025***
			Avg efficiency =		***0.72***	
				1s error =	***0.0012***	
				%error	***0.16***	

SAMPLES

Requested activity units: uCi

Sample quantity units: ml

I.D.	Sample Vol. / mass	Sample units g or mL	Se Recovery	aliquot frac. anal.	Se -79 R.O.I. cpm	ct. error 1 sigma %	*Se-79 Result uCi/ml*	*Se-79 1s TPU uCi/ml*	*Se-79 MDC uCi/ml*	TPU,%
Reag BlK	1.000	mL	0.83	1.00	12.15	140	*2.65E-07*	*3.74E-07*	*1.23E-06*	**141%**
01-1017-PB	1.000	mL	0.77	1.00	16.06	12	*3.48E-06*	*4.71E-07*	*1.33E-06*	**14%**
01-1017	1.000	mL	0.43	1.00	14.14	22	*3.36E-06*	*7.70E-07*	*2.33E-06*	**23%**
01-1017 -dup	1.000	mL	0.68	1.00	12.85	47	*9.65E-07*	*4.67E-07*	*1.49E-06*	**48%**
01-1613	1.000	mL	0.83	1.00	23.87	5	*9.15E-06*	*5.56E-07*	*1.23E-06*	**6%**
01-1842	1.000	mL	0.38	1.00	13.91	24	*3.52E-06*	*8.85E-07*	*2.71E-06*	**25%**
01-1843	1.000	mL	0.63	1.00	12.26	107	*4.56E-07*	*4.92E-07*	*1.61E-06*	**108%**
01-1843-dup	1.000	mL	0.68	1.00	11.52	172	*-2.59E-07*	*4.50E-07*	*1.50E-06*	**174%**

RG Brown 9/12/01

Entered by Date

LR Greenwood 9-13-01

Reviewed by Date

Correction for Hot Cell Fusion									
	Se-79 Result	*Se-79 1s TPU*	*Se-79 MDC*		Hot cell prep		*Se-79 Result*	*Se-79 1s TPU*	*Se-79 MDC*
I.D.	*uCi/ml*	*uCi/ml*	*uCi/ml*	TPU,%	*g*	*ml*	*uCi/g*	*uCi/g*	*uCi/g*
Reag Blk	*2.65E-07*	*3.74E-07*	*1.23E-06*	**141%**	*1.88E-01*	*1.00E+02*	*1.41E-04*	*1.99E-04*	*6.55E-04*
01-1017-PB	*3.48E-06*	*4.71E-07*	*1.33E-06*	**14%**	*1.88E-01*	*1.00E+02*	*1.85E-03*	*2.50E-04*	*7.06E-04*
01-1017	*3.36E-06*	*7.70E-07*	*2.33E-06*	**23%**	*1.84E-01*	*1.00E+02*	*1.83E-03*	*4.19E-04*	*1.27E-03*
01-1017 -dup	*9.65E-07*	*4.67E-07*	*1.49E-06*	**48%**	*1.94E-01*	*1.00E+02*	*4.97E-04*	*2.41E-04*	*7.69E-04*

9/12/01

9-13-01

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

PNL-ALO-115 Solubilization of Metals from Solids Using a KOH-KNO3 Fusion

to 5/24/01

Client name: ~~G Smith~~ R. Hallen
Work Auth. Doc (WAD): ASR 6107
Tank/Core/Project: AN-102 Washed solids HLW feed characterization
Special instructions See ASR Special Instructions

Work package number: W57984
Project number: 42365
PNL QA plan: SBMS
PNL impact level:
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number:

	ACL Sample ID	ACL order number or Client sample ID	Crucible Identifier	Crucible weight (g)	Crucible + sample weight (g)	Sample weight (g)	Spike added Vol. (ml)	Spike added Weight. (g)	Final solution Volume (ml)	Process Factor (1)
1	01-01017-Blank-Ni	Blank				$\bar{x}$ = 0.188			100	
2	01-01017 -Ni	LS-16	1	33.5983	33.7819	0.1836				544.66
3	01-01017 Dup -Ni	LS-16-Dup	2	41.9143	42.1085	0.1942				514.93
4	01-01017-LCS	LS-16-LCS	3	41.7546	41.9416	0.1870				534.76
4	01-01017-BS ICP/MS	LS-16-BS ICP/MS	4	43.6695	—		.1	0.0990		1010.10
5	01-01017-MS ICP/MS	LS-16-MS ICP/MS	5	40.2554	40.4492	0.1938	.1	0.0963		515.99

Analyst's sample preparation comments: DOSE RATE:

BS = 43.7685
MS = 40.5455

Spike source:
PNL spike ID number:
Anal. balance M&TE: 360-06-01-016
HCl volume added (ml): 1
Solution heated (yes/no): yes
Sample filtered (yes/no): NO

(1) Process factor = Final volume (ml) / [Crucible & sample weight (g) - Crucible weight (g)]

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Rev. 2.0 7-28-95 JMR

Analyst/Date: [signature] 5/21/01 Reviewer/Date: [signature] 5/21/01

Battelle, PNNL / AIAL
Inorganic Analysis / ICP-MS Data Report

Project / WP#:	42365 / W57984
ASR#:	6107
Client:	Rich Hallen
Total Samples:	4

RPL #	Client ID
01-1014	LS-12
01-1015	LS-13
01-1016	LS-14
01-1017	LS-16

Procedure: PNL-ALO-280 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number: WB36913 ICP/MS, VG Elemental
512-06-01-014 Mettler AJ100 Balance

Analyst: James P Bramson

Analysis Dates: 9/12/01, 9/25/01, 9/26/01, 9/28/01, 10/1/01

Analysis Files: Experiment - 12SEP01, 25SEP01, 25SEP01b, 26SEP01, 28SEP01, 01OCT01
Procedure - 010912a, 010925a, 010925b, 010926a, 010928a, 011001a
Element Menu - CsTcRb, UNp, Uiso Pu, multi, Tc

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

James Bramson 11/21/02
Analyst

TMP Thomas 11-26-02
Reviewed By

1. Analysis

Four samples, 2 filtrates, 1 wash composite, and 1 solid prepared by the RPG, were submitted for analysis and analyzed on a radioactive material-contained ICP/MS. The filtrates and wash composite were analyzed for Tc-99. The solids were analyzed for total U, U isotopes, Pu-239 and 240, Np-237, and Ru, Rh, and Pd.

See attached ICP/MS data reports for final results and run order for the analytical batch. The final results have been corrected for all client dilutions and laboratory dilutions performed on the sample during analysis. A process blank, blank spike, duplicate, and matrix spike were also submitted and analyzed with the samples. In addition, replicate and post spike analyses were also performed.

The analysis for Ru, Rh, and Pd was difficult due to the isobaric interferences of SrO isotopes and masses common to both Ru and Pd, and the potential mixture of natural and fission yield isotopic abundances. Some of the sample concentrations were also very near detection limit.

2. Quality Control

Duplicate (DUP). The RPDs for all analyses (duplicate and replicate) were < 15%, with the exception of the solids duplicate analysis for Ru, Pd, and Tc-99. The problem with the Ru and Pd was most likely due to the difficulties mentioned above. The Tc-99 concentrations were very near the detection limit.

Matrix Spike (MS). The MS recoveries for all post spike analyses were within 75% to 125% with the exception of Ru. The analytical lab only had MS information for Ru, Rh, and Pd to calculate recovery, none of which met the QC criteria. The reason for the MS failure is suspected to be due to insufficient spiking level, that is, the MS concentration was less than the EQL (10XMDL).

Process Blank (PB) and Blank Spike (BS). The PBs were all below or near detection. The recovery of the blank spikes met the QC criteria for LCS recovery of 80% to 120%.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB and CCB standards are a 1% high purity nitric acid solution used as the diluent for the samples, except for the Pu analysis, where the eluent for the Pu separation was used. The ICB and CCB standards were at or below the instrument detection level except for the Pu analysis, where the standards were above detection level but 2 to 7 orders of magnitude below the sample concentrations.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). Recoveries of the ICV and CCV standards were within 90% to 110%.

Internal Standard (IS). The ISs were within the range of 30% to 120%.

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: JPBramson 2/25/02

Reviewed by: [signature] 2/17/02

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/18/02

Unless otherwise specified; the results are reported µg analyte/g of original sample.

RPL Log-In #	Sample ID	ICP/MS ID	MDL µg/g	Ru µg/g	±	Ru 1SD	Rec/RPD %	MDL µg/g	Rh µg/g	±	Rh 1SD	Rec/RPD %
	1%HNO_3	ICB		< 7.65E-06	±				< 2.33E-06			
	1%HNO_3	CCB1		< 7.13E-06	±	-1.01E-05		2.17E-06	2.23E-06	±	5.86E-06	
	1%HNO_3	CCB2	7.54E-06	1.56E-05	±	3.18E-06		2.30E-06	2.77E-06	±	5.63E-07	
	True Value			**1.00E-04**					**1.00E-04**			
	ICV	ICV		9.38E-05	±	4.53E-06	94%		9.38E-05	±	3.80E-06	94%
	CCV1	CCV1		9.72E-05	±	2.49E-06	97%		9.52E-05	±	1.59E-06	95%
	CCV3	CCV3		1.10E-04	±	1.17E-05	110%		9.30E-05	±	2.63E-07	93%
	0.1ppb Rh, Pd, Ru (Dowex added)	CCV2	7.62E-06	1.02E-04	±	3.89E-07	102%		9.82E-05	±	2.54E-06	93%
01-01017-PB	PROCESS BLK	Sample1		< 4.61E-02	±	-5.83E-02		1.40E-02	< 1.40E-02	±	-7.90E-03	
01-01017-PB	PROCESS BLK post spike	Sample2	5.46E-02	5.54E-01	±	5.87E-02		1.50E-02	5.04E-01	±	1.42E-02	
Post Spike Concentration expected				6.51E-01			85%		6.67E-01			76%
01-01017-BS2	ICP Blank Spike-2	Sample3	4.62E-02	6.65E-01	±	4.46E-02		1.41E-02	6.70E-01	±	3.36E-02	
Spike Concentration expected				6.42E-01			104%		6.42E-01			102%
01-01017	LS-16	Sample4	3.89E+00	9.02E+00	±	1.16E+00		1.19E+00	2.25E+00	±	4.11E-01	
01-01017	LS-16 replicate	Sample5	3.84E+00	8.25E+00	±	7.47E-01	8.9%	1.17E+00	2.45E+00	±	3.35E-01	8%
01-01017-DUP	LS-16-DUP	Sample6	3.84E+00	1.25E+01	±	1.28E+00	32%	1.17E+00	3.54E+00	±	6.59E-01	44%
01-01017	LS-16 post spike	Sample8	4.16E+00	8.32E+01	±	8.27E+00		1.14E+00	4.71E+01	±	1.37E+00	
Post Spike Concentration expected				5.20E+01			143%		5.20E+01			86%
01-01017-MS2	ICP Matrix Spike-2	Sample7	3.82E+00	2.98E+01	±	2.45E+00		1.16E+00	3.53E+00	±	5.34E-01	
Spike Concentration expected				2.23E+00			932%		2.28E+00			56%

MTE: ICP/MS VG (WB36913), Mettler balance 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/28/01
Instrument Filenames: Experiment (28SEP01), Procedure (010928a), Element Menu (multi)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [initials] 2/17/02

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/18/02

Unless otherwise specified; the results are reported μg analyte/g of

RPL Log-In #	Sample ID	ICP/MS ID	MDL $\mu g/g$		Pd $\mu g/g$	±	1SD	Rec/RPD %
	1%HNO_3	ICB		<	6.32E-06			
	1%HNO_3	CCB1		<	5.89E-06			
	1%HNO_3	CCB2		<	6.23E-06			
	True Value				1.00E-04			
	ICV	ICV			9.35E-05	±	5.73E-06	94%
	CCV1	CCV1			8.84E-05	±	3.15E-06	88%
	CCV3	CCV3			9.28E-05	±	2.47E-06	93%
	0.1ppb Rh, Pd, Ru (Dowex added)	CCV2			8.84E-05	±	6.36E-06	88%
01-01017-PB	PROCESS BLK	Sample1		<	3.80E-02	±	-2.35E+00	
01-01017-PB	PROCESS BLK post spike	Sample2	4.06E-02		7.04E-01	±	9.20E-03	
Post Spike Concentration expected					6.51E-01			108%
01-01017-BS2	ICP Blank Spike-2	Sample3	2.48E-02		6.48E-01	±	2.91E-02	
Spike Concentration expected					6.42E-01			101%
01-01017	LS-16	Sample4		<	3.21E+00	±	-4.16E+01	
01-01017	LS-16 replicate	Sample5		<	3.17E+00	±	-2.19E+00	
01-01017-DUP	LS-16-DUP	Sample6	3.17E+00		4.16E+00	±	2.29E+00	
01-01017	LS-16 post spike	Sample8	3.09E+00		6.38E+01	±	2.60E+00	
Post Spike Concentration expected					5.20E+01			123%
01-01017-MS2	ICP Matrix Spike-2	Sample7	3.15E+00	<	3.15E+00	±	-7.98E+00	
Spike Concentration expected					2.28E+00			

MTE: ICP/MS VG (WB36913), Mettler balance 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/28/01
Instrument Filenames: Experiment (28SEP01), Procedure (010928a), Element Menu (multi)

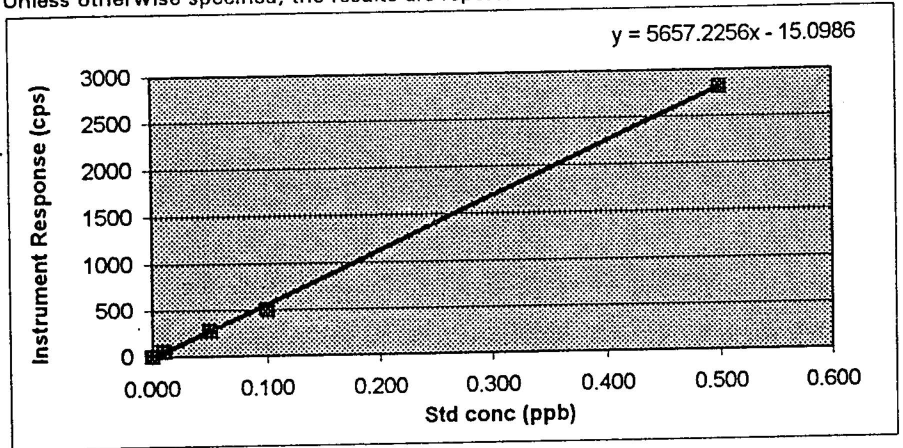
Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: 2/25/02
Reviewed by: 2/17/02

Palladium

Unless otherwise specified; the results are reported in



Slope	5657.2256
Intercept	-15.0986
correl	0.9997

Std (ppb)	(Cts/sec)
0.000	0
0.010	57
0.050	278
0.100	502
0.500	2822

Log-In Number	Sample ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg acps bkg-sub	Std Dev	Conc. µg/g	MDL µg/g	Pd µg/g	±	1SD
	1%HNO3	ICB	1	1	1	0	7	2.67E-06	6.32E-06	6.32E-06	±	1.53E+10
	0.01ppb Rh, Pd, Ru	ICS1	1	1	1	57	20	1.27E-05	6.14E-06	1.27E-05	±	4.43E-06
	0.05ppb Rh, Pd, Ru	ICS2	1	1	1	278	17	5.17E-05	6.14E-06	5.17E-05	±	3.09E-06
	0.1ppb Rh, Pd, Ru	ICS3	1	1	1	502	20	9.14E-05	6.14E-06	9.14E-05	±	3.69E-06
	0.5ppb Rh, Pd, Ru	ICS4	1	1	1	2822	73	5.01E-04	6.20E-06	5.01E-04	±	1.31E-05
	0.1ppb Rh, Pd, Ru	ICV	1	1	1	514	31	9.35E-05	6.21E-06	9.35E-05	±	5.73E-06
	0.1ppb Rh, Pd, Ru	CCV1	1	1	1	485	17	8.84E-05	5.80E-06	8.84E-05	±	3.15E-06
	0.1ppb Rh, Pd, Ru (Dowex adde	CCV2	1	1	1	485	35	8.84E-05	6.29E-06	8.84E-05	±	6.36E-06
	Strontium std	Sr50ppb	1	1	1	5	4	3.60E-06	6.10E-06	6.10E-06	±	4.40E-06
	1%HNO3	CCB1	1	1	1	-3	14	2.16E-06	5.89E-06	5.89E-06	±	-2.84E-05
01-01017-PB	PROCESS BLK	Sample1	6505	260	25	0	14	1.71E-02	3.80E-02	3.80E-02	±	-2.35E+00
01-01017-PB	PROCESS BLK post spike	Sample2	7156	260	27.5	541	7	7.04E-01	4.06E-02	7.04E-01	±	9.20E-03
01-01017-BS2	ICP Blank Spike-2	Sample3	6505	260	25	548	25	6.48E-01	3.81E-02	6.48E-01	±	2.91E-02
01-01017	LS-16	Sample4	520400	260	2000	-1	12	1.31E+00	3.21E+00	3.21E+00	±	-4.16E+01
01-01017	LS-16 replicate	Sample5	520400	260	2000	-12	9	2.40E-01	3.17E+00	3.17E+00	±	-2.19E+00
01-01017-DUP	LS-16-DUP	Sample6	507200	254	2000	31	17	4.16E+00	3.17E+00	4.16E+00	±	2.29E+00
01-01017-MS2	ICP Matrix Spike-2	Sample7	489260	245	2000	-9	21	5.71E-01	3.15E+00	3.15E+00	±	-7.98E+00
01-01017	LS-16 post spike	Sample8	572440	260	2200	615	25	6.38E+01	3.09E+00	6.38E+01	±	2.60E+00
	0.1ppb Rh, Pd, Ru	CCV3	1	1	1	510	14	9.28E-05	6.05E-06	9.28E-05	±	2.47E-06
	Strontium std	Sr100ppb(2)	1	1	1	20	8	6.28E-06	5.99E-06	6.28E-06	±	2.50E-06
	1%HNO3	CCB2	1	1	1	-17	6	-3.38E-07	6.23E-06	6.23E-06	±	-2.23E-06

MTE: ICP/MS VG (WB36913), Mettler balance 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/28/01
Instrument Filenames: Experiment (28SEP01), Procedure (010928a), Element Menu (multi)

Analyst: 2/25/02

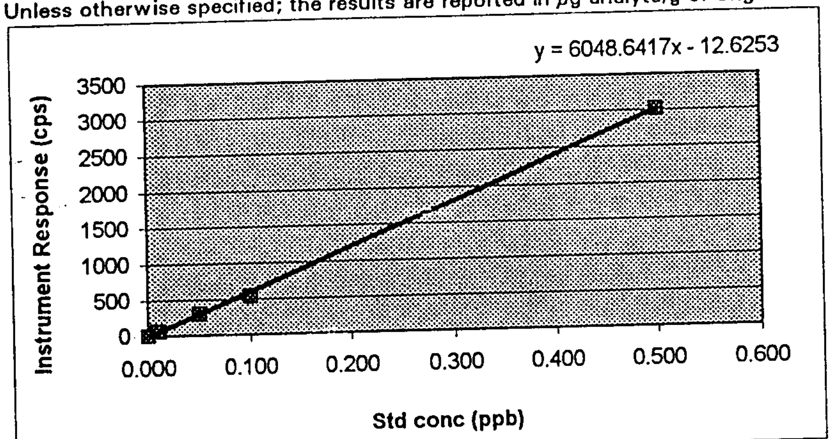
Reviewed By: 02/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R. Hallen
WP/Project: W57984 / 42365
ASR/Log-in: 6107, 01-1017
Report: 2/17/02

Rhodium

Unless otherwise specified; the results are reported in μg analyte/g of original sample.



Slope	6048.6417
Intercept	-12.6253
correl	0.9998

Std (ppb)	(Cts/sec)
0.000	0
0.010	60
0.050	303
0.100	547
0.500	3019

Log-In Number	Sample ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg acps bkg-sub	Std Dev	Conc. $\mu g/g$	MDL $\mu g/g$	Rh 103 $\mu g/g$	±	1SD
	1%HNO3	ICB	1	1	1	0	1	2.09E-06	2.33E-06	2.33E-06	±	-4.48E-06
	0.01ppb Rh, Pd, Ru	ICS1	1	1	1	60	7	1.20E-05	2.26E-06	1.20E-05	±	1.44E-06
	0.05ppb Rh, Pd, Ru	ICS2	1	1	1	303	7	5.21E-05	2.27E-06	5.21E-05	±	1.16E-06
	0.1ppb Rh, Pd, Ru	ICS3	1	1	1	547	2	9.25E-05	2.26E-06	9.25E-05	±	3.35E-07
	0.5ppb Rh, Pd, Ru	ICS4	1	1	1	3019	26	5.01E-04	2.29E-06	5.01E-04	±	4.28E-06
	0.1ppb Rh, Pd, Ru	ICV	1	1	1	555	22	9.38E-05	2.29E-06	9.38E-05	±	3.80E-06
	0.1ppb Rh, Pd, Ru	CCV1	1	1	1	563	9	9.52E-05	2.14E-06	9.52E-05	±	1.59E-06
	0.1ppb Rh, Pd, Ru (Dowex adde	CCV2	1	1	1	581	15	9.82E-05	2.32E-06	9.82E-05	±	2.54E-06
	Strontium std	Sr50ppb	1	1	1	5	1	2.93E-06	2.25E-06	2.93E-06	±	8.57E-07
	1%HNO3	CCB1	1	1	1	1	2	2.23E-06	2.17E-06	2.23E-06	±	5.86E-06
01-01017-PB	PROCESS BLK	Sample1	6505	260	25	-1	1	1.26E-02	1.40E-02	1.40E-02	±	-7.90E-03
01-01017-PB	PROCESS BLK post spike	Sample2	7156	260	27.5	414	12	5.04E-01	1.50E-02	5.04E-01	±	1.42E-02
01-01017-BS2	ICP Blank Spike-2	Sample3	6505	260	25	610	31	6.70E-01	1.41E-02	6.70E-01	±	3.36E-02
01-01017	LS-16	Sample4	520400	260	2000	14	2	2.25E+00	1.19E+00	2.25E+00	±	4.11E-01
01-01017	LS-16 replicate	Sample5	520400	260	2000	16	2	2.45E+00	1.17E+00	2.45E+00	±	3.35E-01
01-01017-DUP	LS-16-DUP	Sample6	507200	254	2000	30	6	3.54E+00	1.17E+00	3.54E+00	±	6.59E-01
01-01017-MS2	ICP Matrix Spike-2	Sample7	489260	245	2000	31	5	3.53E+00	1.16E+00	3.53E+00	±	5.34E-01
01-01017	LS-16 post spike	Sample8	572440	260	2200	485	14	4.71E+01	1.14E+00	4.71E+01	±	1.37E+00
	0.1ppb Rh, Pd, Ru	CCV3	1	1	1	550	2	9.30E-05	2.23E-06	9.30E-05	±	2.63E-07
	Strontium std	Sr100ppb(2)	1	1	1	10	2	3.73E-06	2.21E-06	3.73E-06	±	7.22E-07
	1%HNO3	CCB2	1	1	1	4	1	2.77E-06	2.30E-06	2.77E-06	±	5.63E-07

MTE: ICP/MS VG (WB36913), Mettler balance 512-06-01-014
Analysis Date: 9/28/01
Instrument Filenames: Experiment (28SEP01), Procedure (010928a), Element Menu (multi)

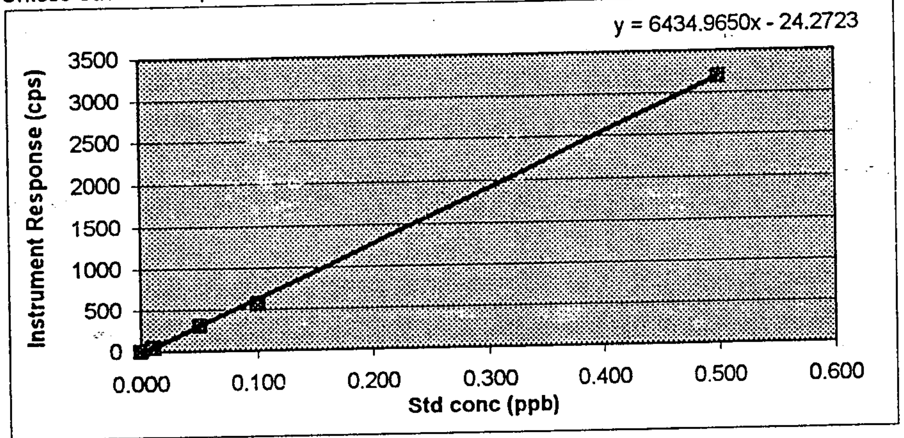
Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: JPBrown 2/25/02
Reviewed by: MPJ 2/17/02

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Ruthenium

Unless otherwise specified; the results are reported in μg analyte/g of original sample.



Slope	6434.9650
Intercept	-24.2723
correl	0.9998

Std (ppb)	(Cts/sec)
0.000	0
0.010	50
0.050	303
0.100	571
0.500	3202

Log-In Number	Sample ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg acps bkg-sub	Std Dev	Conc. μg/g	MDL μg/g		Ru μg/g	±	Ru 1SD
	1%HNO3	ICB	1	1	1	0	16	3.77E-06	7.65E-06	<	7.65E-06	±	#DIV/0!
	0.01ppb Rh, Pd, Ru	ICS1	1	1	1	50	15	1.15E-05	7.43E-06		1.15E-05	±	3.47E-06
	0.05ppb Rh, Pd, Ru	ICS2	1	1	1	303	51	5.09E-05	7.44E-06		5.09E-05	±	8.51E-06
	0.1ppb Rh, Pd, Ru	ICS3	1	1	1	571	26	9.24E-05	7.43E-06		9.24E-05	±	4.15E-06
	0.5ppb Rh, Pd, Ru	ICS4	1	1	1	3202	75	5.01E-04	7.50E-06		5.01E-04	±	1.18E-05
	0.1ppb Rh, Pd, Ru	ICV	1	1	1	579	28	9.38E-05	7.53E-06		9.38E-05	±	4.53E-06
	0.1ppb Rh, Pd, Ru	CCV1	1	1	1	601	15	9.72E-05	7.03E-06		9.72E-05	±	2.49E-06
	0.1ppb Rh, Pd, Ru (Dowex adde	CCV2	1	1	1	635	2	1.02E-04	7.62E-06		1.02E-04	±	3.89E-07
	Strontium std	Sr50ppb	1	1	1	-3	6	3.37E-06	7.39E-06	<	7.39E-06	±	-1.76E-05
	1%HNO3	CCB1	1	1	1	-6	9	2.81E-06	7.13E-06	<	7.13E-06	±	-1.01E-05
01-01017-PB	PROCESS BLK	Sample1	6505	260	25	-6	8	1.84E-02	4.61E-02	<	4.61E-02	±	-5.83E-02
01-01017-PB	PROCESS BLK post spike	Sample2	7156	260	27.5	474	50	5.54E-01	5.46E-02		5.54E-01	±	5.87E-02
01-01017-BS2	ICP Blank Spike-2	Sample3	6505	260	25	634	43	6.65E-01	4.62E-02		6.65E-01	±	4.46E-02
01-01017	LS-16	Sample4	520400	260	2000	87	11	9.02E+00	3.89E+00		9.02E+00	±	1.16E+00
01-01017	LS-16 replicate	Sample5	520400	260	2000	78	7	8.25E+00	3.84E+00		8.25E+00	±	7.47E-01
01-01017-DUP	LS-16-DUP	Sample6	507200	254	2000	134	14	1.25E+01	3.84E+00		1.25E+01	±	1.28E+00
01-01017-MS2	ICP Matrix Spike-2	Sample7	489260	245	2000	367	30	2.98E+01	3.82E+00		2.98E+01	±	2.45E+00
01-01017	LS-16 post spike	Sample8	572440	260	2200	911	91	8.32E+01	4.16E+00		8.32E+01	±	8.27E+00
	0.1ppb Rh, Pd, Ru	CCV3	1	1	1	681	72	1.10E-04	7.33E-06		1.10E-04	±	1.17E-05
	Strontium std	Sr100ppb(2)	1	1	1	90	8	1.78E-05	7.26E-06		1.78E-05	±	1.67E-06
	1%HNO3	CCB2	1	1	1	76	16	1.56E-05	7.54E-06		1.56E-05	±	3.18E-06

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/28/01
Instrument Filenames: Experiment 28SEP01), Procedure(010928a), Element Menu (multi)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Analyst: JJBramson 2/25/02
Reviewed by: 4mR 2.17.02

Unless otherwise specified; the results are reported in μg analyte/g of original sample for total uranium, μCi analyte/ g of original sample for uranium isotopes.

RPL Log-In #	Sample ID	ICP/MS ID	MDL μg/g		Total Uranium μg/g	±	1SD	Rec/RPD %	MDL		U-233 μCi/g	±	1SD	Rec/RPD %
	1%HNO_3	ICB		<	1.39E-04					<	1.34E-06			
	1%HNO_3	CCB1		<	1.37E-04					<	1.32E-06			
	1%HNO_3	CCB2		<	1.52E-04					<	1.47E-06			
	True Value				**2.50E-03**									
	ICV	ICV			2.36E-03	±	2.62E-05	94%						
	CCV1	CCV1			2.37E-03	±	2.24E-05	95%						
	CCV2	CCV2			2.40E-03	±	2.08E-05	96%						
01-01017-PB	PROCESS BLK	Sample1	7.84E-03		8.24E-01	±	5.37E-03		4.51E-09	<	8.10E-06			
01-01017-PB	PROCESS BLK post spike	Sample3	8.45E-03		3.21E+00	±	2.82E-02		1.49E-07	<	1.27E-06			
Post Spike Concentration expected					2.60E+00			92%						
01-01017-BS2	ICP Blank Spike-2	Sample2	7.89E-03		9.08E-01	±	2.02E-02		5.53E-09	<	1.26E-05			
01-01017	LS-16	Sample4	8.29E-02		3.21E+02	±	3.75E+00		3.03E-07		1.17E-03	±	2.31E-05	
01-01017	LS-16 replicate	Sample5	8.84E-02		3.24E+02	±	2.11E+00	1.1%	3.25E-07		1.19E-03	±	4.60E-05	1.7%
01-01017-DUP	LS-16-DUP	Sample6	8.59E-02		3.48E+02	±	4.75E+00	8.1%	3.13E-07		1.27E-03	±	3.34E-05	7.9%
01-01017	LS-16 post spike	Sample8	1.78E-01		5.46E+02	±	4.57E+00		5.59E-07		1.72E-03	±	6.26E-05	
Post Spike Concentration expected					2.08E+02			108%						
01-01017-MS2	ICP Matrix Spike-2	Sample7	8.50E-02		3.07E+02	±	2.90E+00		3.14E-07		1.13E-03	±	2.19E-05	
	1μg =										9.64E-03 μCi			

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01, 25SEP01B), Procedure (010925a, 010925b), Element Menu (UNp, Uiso)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

Unless otherwise specified; the results are reported in μg

RPL Log-In #	Sample ID	ICP/MS ID	MDL	U-234 μCi/g	±	1SD	Rec/RPD %	MDL	U-235 μCi/g	±	1SD	Rec/RPD %
	1%HNO_3	ICB		< 8.67E-07					< 3.01E-10			
	1%HNO_3	CCB1		< 8.53E-07					< 2.96E-10			
	1%HNO_3	CCB2		< 9.48E-07					< 3.29E-10			
	True Value											
	ICV	ICV										
	CCV1	CCV1										
	CCV2	CCV2										
01-01017-PB	PROCESS BLK	Sample1	7.62E-08	< 8.01E-06				1.01E-10	1.06E-08	±	7.39E-09	
01-01017-PB	PROCESS BLK post spike	Sample3	4.76E-09	< 1.81E-06				4.40E-11	1.67E-08	±	2.45E-09	
Post Spike Concentration expected												
01-01017-BS2	ICP Blank Spike-2	Sample2	1.04E-07	< 1.20E-05				5.83E-11	6.71E-09	±	5.75E-09	
01-01017	LS-16	Sample4	3.64E-08	1.41E-04	±	1.07E-05		1.34E-09	5.21E-06	±	7.00E-08	
01-01017	LS-16 replicate	Sample5	3.83E-08	1.40E-04	±	9.06E-06	0.4%	1.44E-09	5.29E-06	±	3.74E-08	1.5%
01-01017-DUP	LS-16-DUP	Sample6	3.78E-08	1.53E-04	±	3.06E-06	8.1%	1.39E-09	5.64E-06	±	6.23E-08	8.0%
01-01017	LS-16 post spike	Sample8	6.37E-08	1.95E-04	±	1.34E-05		2.65E-09	8.13E-06	±	1.56E-07	
Post Spike Concentration expected												
01-01017-MS2	ICP Matrix Spike-2	Sample7	3.69E-08	1.34E-04	±	1.47E-05		1.40E-09	5.06E-06	±	6.09E-08	
	1μg =			6.24E-03 μCi					2.16E-06 μCi			

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01, 25SEP01B), Procedure (010925a, 010925b), Element Menu (UNp, Uiso)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

Unless otherwise specified; the results are reported in μg

RPL Log-In #	Sample ID	ICP/MS ID	MDL	U-236 µCi/g	±	1SD	Rec/RPD %	MDL	U-238 µCi/g	±	1SD	Rec/RPD %
	1%HNO_3	ICB		< 9.00E-09					< 4.68E-11			
	1%HNO_3	CCB1		< 8.86E-09					< 4.60E-11			
	1%HNO_3	CCB2		< 9.85E-09					< 5.11E-11			
	True Value											
	ICV	ICV										
	CCV1	CCV1										
	CCV2	CCV2										
01-01017-PB	PROCESS BLK	Sample1	5.94E-10	< 6.24E-08				2.62E-09	2.76E-07	±	2.15E-09	
01-01017-PB	PROCESS BLK post spike	Sample3	1.66E-11	< 6.30E-09				2.83E-09	1.08E-06	±	8.96E-10	
Post Spike Concentration expected									8.69E-07			92%
01-01017-BS2	ICP Blank Spike-2	Sample2	2.25E-10	< 2.59E-08				2.65E-09	3.05E-07	±	1.42E-09	
01-01017	LS-16	Sample4	2.46E-09	9.53E-06	±	3.65E-07		2.76E-08	1.07E-04	±	8.52E-09	
01-01017	LS-16 replicate	Sample5	2.58E-09	9.45E-06	±	2.54E-07	0.8%	2.95E-08	1.08E-04	±	6.36E-09	1.1%
01-01017-DUP	LS-16-DUP	Sample6	2.38E-09	9.62E-06	±	2.61E-07	0.9%	2.87E-08	1.16E-04	±	1.10E-08	8.1%
01-01017	LS-16 post spike	Sample8	4.48E-09	1.37E-05	±	5.44E-07		5.94E-08	1.82E-04	±	2.11E-08	
Post Spike Concentration expected									6.95E-05			108%
01-01017-MS2	ICP Matrix Spike-2	Sample7	2.50E-09	9.06E-06	±	1.21E-07		2.83E-08	1.02E-04	±	8.93E-09	
	1μg =			6.47E-05 µCi					3.36E-07 µCi			

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01, 25SEP01B), Procedure (010925a, 010925b), Element Menu (UNp, Uiso)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reveiwed by: [initials] 2/11/02

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

RPL Log-In #	Sample ID	ICP/MS ID		Abundance U-233	U-234	U-235	U-236	U-238
	1%HNO_3	ICB	Average	-7.78E-03	4.83E-03	-1.32E-03	-1.37E-02	1.02E+00
			stdev	8.81E-02	1.47E-01	3.60E-01	4.46E-02	4.37E-01
			%RSD	-1132.8%	3051.4%	-27243.6%	-325.1%	42.9%
	NIST U030	ICS1	Average	-2.06E-06	1.90E-04	3.05E-02	2.00E-04	9.69E-01
			stdev	3.10E-06	8.08E-06	1.28E-04	9.14E-06	1.36E-04
			%RSD	-150.2%	4.3%	0.4%	4.6%	0.01%
			True Value		1.90E-04	3.05E-02	2.00E-04	9.69E-01
	NIST 4321B	ICV	Average	-8.44E-07	4.85E-05	7.25E-03	-3.69E-07	9.93E-01
			stdev	1.51E-06	3.75E-06	1.47E-04	1.10E-06	1.48E-04
			%RSD	-178.6%	7.7%	2.0%	-299.3%	0.01%
			True Value		5.29E-05	7.20E-03		9.93E-01
	NIST U030	CCV1	Average	-1.11E-06	1.88E-04	3.04E-02	2.00E-04	9.69E-01
			stdev	2.21E-06	2.89E-06	2.02E-04	7.76E-06	2.04E-04
			%RSD	-199.7%	1.5%	0.7%	3.9%	0.02%
			True Value		1.90E-04	3.05E-02	2.00E-04	9.69E-01
	1%HNO_3	CCB1	Average	3.61E-05	-4.89E-05	2.58E-02	1.95E-04	9.74E-01
			stdev	1.30E-03	1.04E-03	1.07E-02	9.52E-04	1.15E-02
			%RSD	3594.3%	-2120.1%	41.3%	489.2%	1.2%
01-01017-PB	PROCESS BLK	Sample1	Average	-1.02E-03	-1.56E-03	5.98E-03	1.17E-03	9.95E-01
			stdev	2.01E-03	1.40E-03	4.15E-03	2.58E-03	7.78E-03
			%RSD	-196.0%	-90.0%	69.4%	220.5%	0.8%
01-01017-BS2	ICP Blank Spike-2	Sample2	Average	-1.44E-03	-2.12E-03	3.42E-03	4.40E-04	1.00E+00
			stdev	7.15E-04	1.07E-03	2.93E-03	9.41E-04	4.65E-03
			%RSD	-49.8%	-50.6%	85.8%	213.6%	0.5%
01-01017-PB	PROCESS BLK post spike	Sample3	Average	-4.12E-05	-9.04E-05	2.41E-03	-3.03E-05	9.98E-01
			stdev	1.60E-04	1.83E-04	3.53E-04	2.02E-04	8.30E-04
			%RSD	-388.5%	-202.4%	14.6%	-667.2%	0.1%
01-01017	LS-16	Sample4	Average	3.79E-04	7.05E-05	7.51E-03	4.59E-04	9.92E-01
			stdev	7.47E-06	5.37E-06	1.01E-04	1.76E-05	7.90E-05
			%RSD	2.0%	7.6%	1.3%	3.8%	0.01%
01-01017	LS-16 replicate	Sample5	Average	3.81E-04	6.94E-05	7.54E-03	4.50E-04	9.92E-01
			stdev	1.47E-05	4.48E-06	5.33E-05	1.21E-05	5.83E-05
			%RSD	3.9%	6.5%	0.7%	2.7%	0.01%
01-01017-DUP	LS-16-Dup	Sample6	Average	3.78E-04	7.05E-05	7.50E-03	4.27E-04	9.92E-01
			stdev	9.96E-06	1.41E-06	8.28E-05	1.16E-05	9.44E-05
			%RSD	2.6%	2.0%	1.1%	2.7%	0.01%
01-01017-MS2	ICP Matrix Spike-2	Sample7	Average	3.83E-04	6.97E-05	7.62E-03	4.55E-04	9.91E-01
			stdev	7.39E-06	7.66E-06	9.17E-05	6.09E-06	8.64E-05
			%RSD	1.9%	11.0%	1.2%	1.3%	0.01%
01-01017	LS-16 post spike	Sample8	Average	3.26E-04	5.74E-05	6.89E-03	3.89E-04	9.92E-01
			stdev	1.19E-05	3.93E-06	1.32E-04	1.54E-05	1.15E-04
			%RSD	3.6%	6.9%	1.9%	4.0%	0.01%
	NIST U030	CCV2	Average	-1.59E-06	1.83E-04	3.03E-02	2.10E-04	9.69E-01
			stdev	2.63E-06	7.48E-06	2.10E-04	9.99E-06	2.14E-04
			%RSD	-165.2%	4.1%	0.7%	4.8%	0.02%
			True Value		1.90E-04	3.05E-02	2.00E-04	9.69E-01
	1%HNO_3	CCB2	Average	-3.42E-04	-9.23E-04	3.11E-02	-4.02E-04	9.71E-01
			stdev	9.74E-03	1.01E-02	8.35E-03	1.11E-02	3.42E-02
			%RSD	-2847.2%	-1094.1%	26.9%	-2756.0%	3.5%

MTE: ICP/MS VG (WB36913), Mettler balance 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

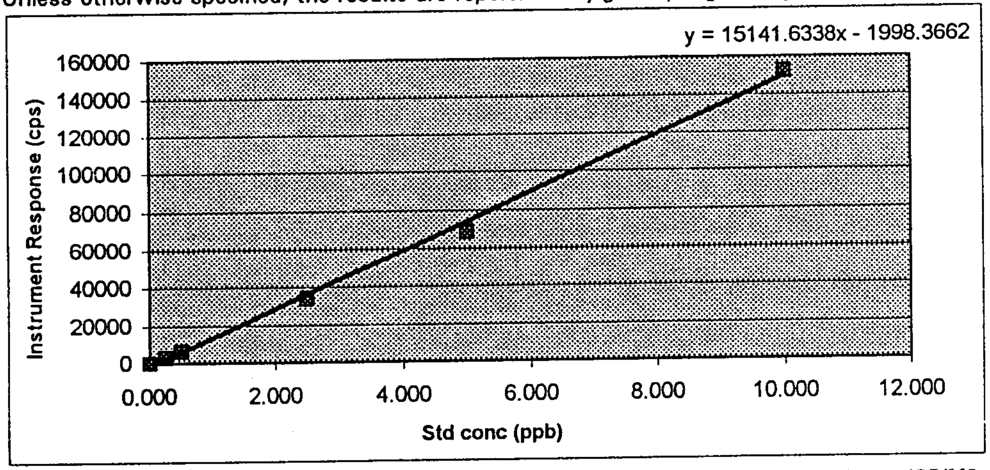
Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/9/02

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2-9-02

Total Uranium

Unless otherwise specified; the results are reported in µg analyte/g of original sample.



Slope	15141.6338
Intercept	-1998.3662
correl	0.9987

Std (ppb)	(Cts/sec)
0.000	0
0.250	3203
0.500	6278
2.500	33765
5.000	68749
10.000	152350

RPL Log-In Number	Sample ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg acps bkg-sub	Std Dev	Conc. µg/g	MDL µg/g	total U µg/g	±	1SD
	1%HNO3	ICB	1	1	1	0	5	1..32E-04	1.39E-04	< 1.39E-04	±	3.90E-06
	0.25ppb U	ICS1	1	1	1	3203	7	3.43E-04	1.38E-04	3.43E-04	±	7.23E-07
	0.5ppb U	ICS2	1	1	1	6278	67	5.47E-04	1.37E-04	5.47E-04	±	5.82E-06
	2.5ppb U	ICS3	1	1	1	33765	544	2.36E-03	1.48E-04	2.36E-03	±	3.81E-05
	5ppb U	ICS4	1	1	1	68749	686	4.67E-03	1.40E-04	4.67E-03	±	4.66E-05
	10ppb U	ICS5	1	1	1	152350	1126	1.02E-02	1.28E-04	1.02E-02	±	7.53E-05
	2.5ppb U	ICV	1	1	1	33691	374	2.36E-03	1.39E-04	2.36E-03	±	2.62E-05
	2.5ppb U	CCV1	1	1	1	33953	320	2.37E-03	1.35E-04	2.37E-03	±	2.24E-05
	1%HNO3	CCB1	1	1	1	-142	3	1.23E-04	1.37E-04	< 1.37E-04	±	-2.60E-06
01-01017-PB	PROCESS BLK	Sample1	5204	260.2	20	398	3	8.24E-01	7.05E-01	8.24E-01	±	5.37E-03
01-01017-BS2	ICP Blank Spike-2	Sample2	5204	260.2	20	644	14	9.08E-01	7.10E-01	9.08E-01	±	2.02E-02
01-01017-PB	PROCESS BLK post spike	Sample3	5204	260.2	20	7341	64	3.21E+00	7.60E-01	3.21E+00	±	2.82E-02
01-01017	LS-16	Sample4	52040	260.2	200	91332	1068	3.21E+02	7.46E+00	3.21E+02	±	3.75E+00
01-01017	LS-16 replicate	Sample5	52040	260.2	200	92399	602	3.24E+02	7.96E+00	3.24E+02	±	2.11E+00
01-01017-DUP	LS-16-DUP	Sample6	50720	253.6	200	101886	1392	3.48E+02	7.73E+00	3.48E+02	±	4.75E+00
01-01017-MS2	ICP Matrix Spike-2	Sample7	48926	244.6	200	93148	877	3.07E+02	7.65E+00	3.07E+02	±	2.90E+00
01-01017	LS-16 post spike	Sample8	104080	260.2	400	77431	648	5.46E+02	1.60E+01	5.46E+02	±	4.57E+00
	2.5ppb U	CCV2	1	1	1	34365	298	2.40E-03	1.53E-04	2.40E-03	±	2.08E-05
	1%HNO3	CCB2	1	1	1	-130	3	1.23E-04	1.52E-04	1.52E-04	±	-3.95E-06

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01), Procedure (010925a), Element Menu (UNp)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	10051.09	1.23	1.77	3.07	1.15	34.09	1.54	-5.42E-06	7.15E-06	1.56E-04	4.91E-06	9.59E-05
2	9576.75	1.07	1.31	1.23	1.07	34.70	0.61	7.04E-05	5.46E-05	6.77E-05	8.95E-05	4.17E-04
3	9313.78	1.15	2.07	1.54	0.84	23.95	2.15	-7.93E-05	-2.29E-05	-6.13E-05	-9.50E-05	-8.02E-04
4	9045.28	1.77	1.92	0.92	1.23	32.25	1.54	4.86E-05	2.60E-05	-6.32E-05	9.07E-06	2.52E-04
5	8892.29	1.31	1.38	0.92	1.38	30.10	1.84	-3.42E-05	-6.49E-05	-9.86E-05	-8.43E-06	3.45E-05
					Average Bkg:		1.54					
					avg		1.54E+00	3.96E-09	-2.46E-09	6.73E-10	6.98E-09	-5.19E-07
					Std Dev		5.14E-01	5.44E-05	4.11E-05	9.62E-05	5.87E-05	4.22E-04
					%RSD		33.47	1372909.21	-1668015.93	14296530.44	839985.97	-81413.33

Spike Factor 233:	1.000000	2.62E+12
Spike Factor 234:		
Spike Factor 235:		
Spike Factor 236:		
Spike Factor 238:		

Atomic Weight 233:	233
Atomic Weight 234:	234.0409
Atomic Weight 235:	235.0439
Atomic Weight 236:	236.0000
Atomic Weight 238:	238.0508

SAMPLE NAME	ICB - 1%HNO_3
Sample Weight:	
NG OF 230 ADDED:	1.00
Volume Submitted:	

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	1.04E+04	-7.776E-03
U234 =	-6.45E+03	4.833E-03
U235 =	1.76E+03	-1.321E-03
U236 =	1.83E+04	-1.371E-02
U238 =	-1.36E+06	1.018E+00
Total U	-1.33E+06	1.00E+00

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/11/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	7654.29	1.07	67.64	9828.36	65.49	309392.78	2.15	-1.11E-04	8.23E-03	1.27E+00	8.15E-03	4.04E+01
2	8121.53	1.07	61.27	9364.16	66.41	295146.31	1.54	-3.06E-05	7.07E-03	1.14E+00	7.87E-03	3.64E+01
3	8145.49	1.46	56.51	9118.40	62.03	285464.50	1.54	1.49E-05	6.49E-03	1.11E+00	7.32E-03	3.51E+01
4	8102.79	1.61	60.88	8811.50	61.57	277110.69	1.84	-3.37E-06	7.01E-03	1.08E+00	7.27E-03	3.42E+01
5	8010.32	1.38	57.89	8597.68	56.89	272361.94	3.69	-2.53E-04	6.51E-03	1.06E+00	6.55E-03	3.40E+01
					Average Bkg:		2.15					
					avg		2.15E+00	-7.67E-05	7.06E-03	1.13E+00	7.43E-03	3.60E+01
					Std Dev		8.01E-01	9.82E-05	6.33E-04	7.48E-02	5.52E-04	2.36E+00
					%RSD		37.25	-128.03	8.97	6.60	7.43	6.55

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME	ICS1 - U030
Sample Weight:	
NG OF 230 ADDED:	1.00
Volume Submitted:	

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance	certified
U233 =	-2.01E+08	-2.064E-06	
U234 =	1.85E+10	1.900E-04	1.90E-04
U235 =	2.97E+12	3.050E-02	3.05E-02
U236 =	1.95E+10	2.000E-04	2.00E-04
U238 =	9.43E+13	9.691E-01	9.69E-01
Total U	9.73E+13	1.00E+00	

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [initials] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	11754.99	1.38	22.19	3004.49	1.77	396127.38	2.46	-6.41E-05	1.60E-03	2.53E-01	-1.53E-05	3.37E+01
2	9022.24	1.00	21.96	2717.02	1.31	375348.94	1.54	-3.34E-05	2.17E-03	2.98E-01	1.73E-05	4.16E+01
3	8422.89	1.61	20.58	2690.30	1.07	365543.50	1.23	6.79E-05	2.20E-03	3.17E-01	-2.45E-05	4.34E+01
4	8062.24	1.07	18.12	2578.20	1.00	356116.50	2.15	-1.04E-04	1.89E-03	3.17E-01	-9.77E-05	4.42E+01
5	7797.75	1.61	20.73	2592.02	1.77	346693.12	2.15	-4.24E-05	2.28E-03	3.29E-01	-5.90E-06	4.45E+01
Average Bkg:							1.90					
avg							1.90E+00	-3.53E-05	2.03E-03	3.03E-01	-1.54E-05	4.15E+01
Std Dev							4.51E-01	5.71E-05	2.50E-04	2.67E-02	4.36E-05	4.01E+00
%RSD							23.70	-161.95	12.30	8.82	-283.22	9.67

Spike Factor 233:	1.000000	2.62E+12
Spike Factor 234:		
Spike Factor 235:		
Spike Factor 236:		
Spike Factor 238:		

Atomic Weight 233:	233
Atomic Weight 234:	234.0409
Atomic Weight 235:	235.0439
Atomic Weight 236:	236.0000
Atomic Weight 238:	238.0508

SAMPLE NAME ICV - NIST 4321b
Sample Weight:
NG OF 230 ADDED: 1.00
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance	Abundance certified
U233 =	-9.24E+07	-8.443E-07	
U234 =	5.31E+09	4.854E-05	5.29E-05
U235 =	7.93E+11	7.247E-03	7.20E-03
U236 =	-4.03E+07	-3.686E-07	
U238 =	1.09E+14	9.927E-01	9.93E-01
Total U	1.09E+14	1.00E+00	

Analytical Equipment: ICP/MS VG WB3691, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	7242.05	1.61	61.04	9416.08	65.80	300726.84	1.84	-6.63E-06	7.86E-03	1.29E+00	8.70E-03	4.15E+01
2	7776.55	1.61	59.27	8897.51	60.27	279802.47	2.46	-8.06E-05	7.03E-03	1.13E+00	7.33E-03	3.60E+01
3	7841.06	1.38	57.58	8618.88	56.04	271733.44	2.46	-1.08E-04	6.76E-03	1.09E+00	6.74E-03	3.47E+01
4	7858.26	1.07	55.35	8476.34	57.89	266926.19	0.61	8.05E-05	6.70E-03	1.07E+00	7.18E-03	3.40E+01
5	7753.82	0.92	53.59	8223.52	58.12	258122.25	1.84	-9.05E-05	6.42E-03	1.05E+00	7.16E-03	3.33E+01
					Average Bkg:		1.84					
					avg		1.84E+00	-4.11E-05	6.95E-03	1.13E+00	7.42E-03	3.59E+01
					Std Dev		6.73E-01	7.00E-05	4.94E-04	8.57E-02	6.67E-04	2.96E+00
					%RSD		36.51	-170.32	7.11	7.61	8.99	8.25

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME CCV1 - U030
Sample Weight:
NG OF 230 ADDED: 1.00
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance	Abundance certified
U233 =	-1.08E+08	-1.109E-06	
U234 =	1.82E+10	1.877E-04	1.90E-04
U235 =	2.95E+12	3.042E-02	3.05E-02
U236 =	1.94E+10	2.003E-04	2.00E-04
U238 =	9.40E+13	9.692E-01	9.69E-01
Total U	9.70E+13	1.00E+00	

Analytical Equipment: ICP/MS VG WB36913
Balance, Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/9/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	11935.34	1.07	2.30	160.00	1.84	4914.93	1.54	-1.32E-05	4.71E-05	1.32E-02	6.76E-05	4.09E-01
2	9106.11	1.92	0.84	8.29	0.61	560.76	1.23	9.71E-05	-5.56E-05	7.73E-04	-2.38E-05	5.83E-02
3	8386.02	1.00	2.07	6.76	1.69	336.58	1.84	-7.30E-05	1.15E-05	5.85E-04	2.44E-05	3.68E-02
4	8043.19	0.84	1.23	4.30	1.07	295.43	0.92	1.48E-05	2.19E-05	4.20E-04	6.10E-05	3.35E-02
5	7710.20	1.61	1.54	2.15	1.38	245.68	1.84	-4.77E-06	-5.33E-05	4.36E-05	-1.62E-05	2.85E-02
					Average Bkg:		1.47					
					avg		1.47E+00	4.20E-06	-5.68E-06	3.00E-03	2.26E-05	1.13E-01
					Std Dev		3.58E-01	5.50E-05	4.15E-05	5.09E-03	3.78E-05	1.48E-01
					%RSD		24.30	1309.35	-729.61	169.80	167.36	130.89

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME CCB1 - 1% HNO_3

Sample Weight:
NG OF 230 ADDED: 1.00
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	1.10E+07	3.612E-05
U234 =	-1.49E+07	-4.890E-05
U235 =	7.85E+09	2.580E-02
U236 =	5.92E+07	1.946E-04
U238 =	2.96E+11	9.740E-01
Total U	3.04E+11	1.00E+00

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: tmf 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	9326.68	1.69	1.23	5.83	2.38	536.81	0.61	1.35E-04	4.85E-05	5.59E-04	2.28E-04	5.44E-02
2	8813.65	1.38	1.84	6.45	1.07	526.37	2.76	-1.27E-04	-1.16E-04	4.19E-04	-1.45E-04	5.63E-02
3	8457.60	1.07	1.69	2.15	3.15	475.08	2.46	-1.33E-04	-1.02E-04	-3.19E-05	1.22E-04	5.28E-02
4	8315.68	1.54	1.23	4.61	3.15	501.18	2.46	-8.28E-05	-1.57E-04	2.60E-04	1.24E-04	5.69E-02
5	8151.63	1.31	1.38	5.83	1.77	483.37	2.15	-7.58E-05	-1.06E-04	4.52E-04	-3.81E-06	5.59E-02
					Average Bkg:		2.09					
					avg		2.09E+00	-5.68E-05	-8.65E-05	3.32E-04	6.50E-05	5.52E-02
					Std Dev		7.62E-01	9.87E-05	7.03E-05	2.05E-04	1.28E-04	1.49E-03
					%RSD		36.50	-173.64	-81.27	61.94	197.53	2.70

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME 01-01017, Process Blank
Sample Weight:
NG OF 230 ADDED: 1.00
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	-1.49E+08	-1.024E-03
U234 =	-2.27E+08	-1.559E-03
U235 =	8.69E+08	5.976E-03
U236 =	1.70E+08	1.170E-03
U238 =	1.45E+11	9.954E-01
Total U	1.45E+11	1.00E+00

Analytical Equipment: ICP/MS VG WB36913,
Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	8908.27	1.07	2.07	17.20	1.69	6363.21	0.92	4.06E-05	1.10E-04	1.82E-03	1.27E-04	7.11E-01
2	8308.30	1.46	1.15	16.58	1.46	5955.28	3.07	-1.63E-04	-2.38E-04	1.62E-03	-1.48E-04	7.14E-01
3	7966.40	1.54	0.84	16.28	1.31	5815.83	0.92	9.83E-05	-2.42E-05	1.92E-03	8.96E-05	7.27E-01
4	7861.64	1.46	1.38	17.20	1.38	5664.40	1.54	1.46E-05	-3.38E-05	1.98E-03	2.32E-05	7.18E-01
5	7738.15	1.15	1.46	12.90	0.54	5577.16	2.46	-1.39E-04	-1.39E-04	1.34E-03	-2.01E-04	7.18E-01
					Average Bkg:		1.78					
					avg		1.78E+00	-2.96E-05	-6.50E-05	1.73E-03	-2.18E-05	7.18E-01
					Std Dev		8.55E-01	1.03E-04	1.17E-04	2.31E-04	1.30E-04	5.37E-03
					%RSD		48.03	-347.52	-179.90	13.33	-595.68	0.75

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME 01-01017, Process Blank post spike
Sample Weight:
NG OF 230 ADDED: 1.00
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	-7.75E+07	-4.116E-05
U234 =	-1.70E+08	-9.043E-05
U235 =	4.54E+09	2.411E-03
U236 =	-5.71E+07	-3.033E-05
U238 =	1.88E+12	9.978E-01
Total U	1.88E+12	1.00E+00

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: 2/25/02
Reviewed by: 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	9452.95	1.46	1.07	4.91	2.30	753.62	2.15	-4.64E-05	-1.25E-04	2.94E-04	5.83E-05	7.64E-02
2	8723.95	2.07	0.77	3.99	2.23	693.74	3.38	-1.20E-04	-3.03E-04	7.39E-05	-8.70E-05	7.60E-02
3	8488.94	1.46	1.38	7.98	2.99	691.28	2.46	-8.93E-05	-1.37E-04	6.50E-04	1.04E-04	7.81E-02
4	8436.71	0.84	1.61	4.30	2.69	677.15	2.76	-1.95E-04	-1.46E-04	1.85E-04	3.34E-05	7.68E-02
5	8166.99	1.07	1.38	3.07	2.30	665.48	2.15	-1.03E-04	-1.06E-04	1.16E-04	6.08E-05	7.81E-02
					Average Bkg:		2.58					
					avg		2.58E+00	-1.11E-04	-1.63E-04	2.64E-04	3.40E-05	7.71E-02
					Std Dev		4.60E-01	4.88E-05	7.13E-05	2.07E-04	6.47E-05	8.51E-04
					%RSD		17.82	-44.01	-43.65	78.49	190.42	1.10

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME 01-01017-BS2, ICP Blank Spike-2
Sample Weight:
NG OF 230 ADDED: 1.00
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	-2.90E+08	-1.437E-03
U234 =	-4.28E+08	-2.118E-03
U235 =	6.90E+08	3.419E-03
U236 =	8.89E+07	4.404E-04
U238 =	2.02E+11	9.997E-01
Total U	2.02E+11	1.00E+00

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/19/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	9197.66	118.54	25.18	2348.77	138.19	301574.75	2.76	1.22E-02	2.33E-03	2.53E-01	1.45E-02	3.28E+01
2	8329.19	113.16	24.34	2151.60	140.27	284437.19	1.54	1.29E-02	2.62E-03	2.56E-01	1.64E-02	3.42E+01
3	8041.04	112.78	23.26	2106.15	133.20	277300.31	1.23	1.34E-02	2.63E-03	2.60E-01	1.61E-02	3.45E+01
4	7689.92	110.09	20.96	2083.73	124.60	269780.59	1.84	1.36E-02	2.38E-03	2.68E-01	1.57E-02	3.51E+01
5	7441.10	107.10	19.19	1991.90	130.13	263685.66	1.54	1.37E-02	2.27E-03	2.65E-01	1.70E-02	3.55E+01
					Average Bkg:		1.78					
					avg		1.78E+00	1.32E-02	2.45E-03	2.60E-01	1.59E-02	3.44E+01
					Std Dev		5.28E-01	5.63E-04	1.49E-04	5.73E-03	8.38E-04	9.17E-01
					%RSD		29.66	4.28	6.08	2.20	5.26	2.67

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME **01-01017, LS-16**
Sample Weight:
NG OF 230 ADDED: **1.00**
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	3.44E+10	3.791E-04
U234 =	6.41E+09	7.053E-05
U235 =	6.82E+11	7.505E-03
U236 =	4.17E+10	4.590E-04
U238 =	9.01E+13	9.916E-01
Total U	9.09E+13	1.00E+00

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	8358.99	123.07	23.80	2272.91	137.43	296827.53	1.84	1.40E-02	2.52E-03	2.69E-01	1.59E-02	3.55E+01
2	7832.77	111.48	22.80	2126.42	128.52	277752.66	2.15	1.35E-02	2.53E-03	2.69E-01	1.59E-02	3.55E+01
3	7491.79	114.01	19.65	2051.48	125.83	269691.25	2.15	1.44E-02	2.24E-03	2.71E-01	1.62E-02	3.60E+01
4	7385.19	105.79	21.04	2025.37	127.68	261775.77	0.92	1.37E-02	2.61E-03	2.72E-01	1.69E-02	3.55E+01
5	7043.61	98.27	21.19	1985.76	119.00	257150.44	1.84	1.32E-02	2.63E-03	2.79E-01	1.63E-02	3.65E+01
					Average Bkg:		1.78					
					avg		1.78E+00	1.38E-02	2.50E-03	2.72E-01	1.62E-02	3.58E+01
					Std Dev		4.51E-01	4.15E-04	1.41E-04	3.74E-03	3.57E-04	4.17E-01
					%RSD		25.34	3.02	5.64	1.37	2.20	1.16

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME	01-01017, LS-16 replicate
Sample Weight:	
NG OF 230 ADDED:	1.00
Volume Submitted:	

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	3.60E+10	3.812E-04
U234 =	6.56E+09	6.936E-05
U235 =	7.13E+11	7.537E-03
U236 =	4.25E+10	4.499E-04
U238 =	9.38E+13	9.916E-01
Total U	9.46E+13	1.00E+00

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/0.
Reviewed by: [signature] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	8053.33	129.59	25.95	2461.49	141.96	323379.12	2.46	1.52E-02	2.80E-03	3.03E-01	1.70E-02	4.02E+01
2	7508.99	124.60	23.88	2309.77	134.20	304509.56	1.54	1.58E-02	2.85E-03	3.05E-01	1.74E-02	4.06E+01
3	7201.19	112.70	24.03	2222.24	132.28	293932.47	1.54	1.49E-02	3.00E-03	3.06E-01	1.78E-02	4.08E+01
4	6988.62	115.85	21.73	2237.29	131.36	287831.75	0.31	1.60E-02	2.94E-03	3.17E-01	1.84E-02	4.12E+01
5	6826.43	114.16	22.34	2146.38	121.07	281406.69	1.54	1.59E-02	2.92E-03	3.12E-01	1.72E-02	4.12E+01
					Average Bkg:		1.47					
					avg		1.47E+00	1.56E-02	2.90E-03	3.08E-01	1.76E-02	4.08E+01
					Std Dev		6.84E-01	4.22E-04	6.93E-05	5.31E-03	5.04E-04	3.99E-01
					%RSD		46.40	2.71	2.39	1.72	2.87	0.98

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME **01-01017-Dup, LS-16 DUP**
Sample Weight:
NG OF 230 ADDED: **1.00**
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	4.08E+10	3.783E-04
U234 =	7.60E+09	7.050E-05
U235 =	8.08E+11	7.495E-03
U236 =	4.60E+10	4.269E-04
U238 =	1.07E+14	9.916E-01
Total U	1.08E+14	1.00E+00

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	7914.79	130.67	27.10	2412.66	145.18	314716.81	0.61	1.59E-02	3.21E-03	3.02E-01	1.79E-02	3.98E+01
2	7322.53	121.69	23.26	2313.45	144.10	295815.94	3.69	1.56E-02	2.56E-03	3.13E-01	1.88E-02	4.04E+01
3	7064.49	115.47	22.88	2227.46	137.20	288415.41	2.15	1.55E-02	2.81E-03	3.12E-01	1.88E-02	4.09E+01
4	7009.20	114.16	23.26	2181.70	131.82	282939.59	0.92	1.56E-02	3.06E-03	3.08E-01	1.83E-02	4.04E+01
5	6762.23	112.24	20.96	2183.23	132.97	276068.19	2.76	1.56E-02	2.58E-03	3.20E-01	1.89E-02	4.09E+01
					Average Bkg:		2.03					
					avg		2.03E+00	1.56E-02	2.84E-03	3.11E-01	1.86E-02	4.05E+01
					Std Dev		1.14E+00	1.26E-04	2.57E-04	5.79E-03	3.70E-04	3.95E-01
					%RSD		56.37	0.81	9.03	1.86	1.99	0.98

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME 01-01017, ICP Matrix Spike-2
Sample Weight:
NG OF 230 ADDED: 1.00
Volume Submitted:

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	4.09E+10	3.829E-04
U234 =	7.45E+09	6.970E-05
U235 =	8.15E+11	7.624E-03
U236 =	4.86E+10	4.549E-04
U238 =	1.06E+14	9.915E-01
Total U	1.07E+14	1.00E+00

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 10/2/01

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: JF Bryan 2/25/02
Reviewed by: 2/11/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	3847.89	54.05	10.44	1119.69	62.95	157207.38	1.54	1.32E-02	2.22E-03	2.88E-01	1.57E-02	4.09E+01
2	3536.14	52.59	9.52	1023.26	60.11	147461.03	0.92	1.41E-02	2.33E-03	2.87E-01	1.64E-02	4.17E+01
3	3447.69	49.60	11.36	1030.02	57.50	144433.66	3.07	1.30E-02	2.30E-03	2.96E-01	1.55E-02	4.19E+01
4	3413.90	49.90	10.52	1000.23	60.65	141225.23	1.54	1.37E-02	2.52E-03	2.90E-01	1.70E-02	4.14E+01
5	3301.80	49.44	9.75	944.34	56.97	138713.38	0.61	1.43E-02	2.65E-03	2.83E-01	1.68E-02	4.20E+01
					Average Bkg:		1.54					
					avg		1.54E+00	1.37E-02	2.40E-03	2.89E-01	1.63E-02	4.16E+01
					Std Dev		8.47E-01	4.89E-04	1.59E-04	4.03E-03	5.88E-04	4.18E-01
					%RSD		55.14	3.58	6.60	1.40	3.61	1.00

Spike Factor 233:	1.000000	2.62E+12
Spike Factor 234:		
Spike Factor 235:		
Spike Factor 236:		
Spike Factor 238:		

Atomic Weight 233:	233
Atomic Weight 234:	234.0409
Atomic Weight 235:	235.0439
Atomic Weight 236:	236.0000
Atomic Weight 238:	238.0508

SAMPLE NAME	01-01017, LS-16 post spike
Sample Weight:	
NG OF 230 ADDED:	1.00
Volume Submitted:	

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance
U233 =	3.58E+10	3.258E-04
U234 =	6.30E+09	5.736E-05
U235 =	7.56E+11	6.890E-03
U236 =	4.27E+10	3.886E-04
U238 =	1.09E+14	9.923E-01
Total U	1.10E+14	1.00E+00

Analytical Equipment: ICP/MS VG WB36913, Mettler balance 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/02
Reviewed by: [signature] 2/17/02

SAMPLE / CHECK ISOTOPIC CALCULATION

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Ratio U233/Th230	Ratio U234/Th230	Ratio U235/Th230	Ratio U236/Th230	Ratio U238/Th230
1	6492.53	1.46	54.13	8539.62	63.03	270825.16	1.54	1.26E-05	7.79E-03	1.30E+00	9.33E-03	4.17E+01
2	6747.79	1.77	51.75	7955.03	56.81	254589.72	1.84	1.31E-05	7.11E-03	1.17E+00	8.03E-03	3.78E+01
3	6757.62	0.92	53.74	7879.77	52.97	248454.56	2.46	-1.95E-04	7.30E-03	1.16E+00	7.37E-03	3.68E+01
4	6897.70	2.00	51.28	7730.47	57.96	242702.47	2.15	2.57E-06	6.85E-03	1.11E+00	7.97E-03	3.52E+01
5	6751.48	1.00	46.22	7507.45	54.66	237489.66	2.15	-1.40E-04	6.28E-03	1.10E+00	7.67E-03	3.52E+01
					Average Bkg:		2.03					
					avg		2.03E+00	-6.14E-05	7.07E-03	1.17E+00	8.07E-03	3.73E+01
					Std Dev		3.13E-01	8.85E-05	5.01E-04	7.24E-02	6.69E-04	2.41E+00
					%RSD		15.45	-144.15	7.09	6.19	8.29	6.44

Spike Factor 233:	1.000000	2.62E+12	Atomic Weight 233:	233
Spike Factor 234:			Atomic Weight 234:	234.0409
Spike Factor 235:			Atomic Weight 235:	235.0439
Spike Factor 236:			Atomic Weight 236:	236.0000
Spike Factor 238:			Atomic Weight 238:	238.0508

SAMPLE NAME	CCV2 - U030
Sample Weight:	
NG OF 230 ADDED:	1.00
Volume Submitted:	

ISOTOPE DILUTION ANALYSIS CALCULATIONS

	atoms	Abundance	certified
U233 =	-1.61E+08	-1.595E-06	
U234 =	1.85E+10	1.834E-04	1.90E-04
U235 =	3.06E+12	3.033E-02	3.05E-02
U236 =	2.11E+10	2.096E-04	2.00E-04
U238 =	9.78E+13	9.693E-01	9.69E-01
Total U	1.01E+14	1.00E+00	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/17/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [illegible] 2/25/02
Reveiwed by: [illegible] 2/17/02

ICB

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Isotopic Abundance U233/Th230	Isotopic Abundance U234/Th230	Isotopic Abundance U235/Th230	Isotopic Abundance U236/Th230	Isotopic Abundance U238/Th230
1	10051.09	1.23	1.77	3.07	1.15	34.09	1.54	-3.06E-05	2.29E-05	1.5E-04	-3.82E-05	3.24E-03
2	9576.75	1.07	1.31	1.23	1.07	34.70	0.61	4.81E-05	7.22E-05	6.4E-05	4.81E-05	3.56E-03
3	9313.78	1.15	2.07	1.54	0.84	23.95	2.15	-1.07E-04	-8.24E-06	-6.6E-05	-1.40E-04	2.34E-03
4	9045.28	1.77	1.92	0.92	1.23	32.25	1.54	2.55E-05	4.24E-05	-6.8E-05	-3.40E-05	3.40E-03
5	8892.29	1.31	1.38	0.92	1.38	30.10	1.84	-6.04E-05	-5.18E-05	-1.0E-04	-5.18E-05	3.18E-03
							1.54	-2.492E-05	1.549E-05	-4.114E-06	-4.321E-05	3.143E-03
						std	5.14E-01	5.64E-05	4.26E-05	9.70E-05	5.99E-05	4.22E-04
						%rsd	33.47	-226.49	275.16	-2358.42	-138.57	13.43

ISOTOPE RATIO MASS BIAS CORRECTION

Sample number: UO3O

Run No.	Raw Counts 230	Raw Counts 233	Raw Counts 234	Raw Counts 235	Raw Counts 236	Raw Counts 238	Raw Counts 226	Isotopic Ratio U233/Th230	Isotopic Ratio U234/Th230	Isotopic Ratio U235/Th230	Isotopic Ratio U236/Th230	Isotopic Ratio U238/Th230
1	7654.29	1.07	67.64	9828.36	65.49	309392.78	2.15	-1.16E-04	8.54E-03	1.28E+00	8.32E-03	4.04E+01
2	8121.53	1.07	61.27	9364.16	66.41	295146.31	1.54	-3.18E-05	7.34E-03	1.15E+00	8.03E-03	3.63E+01
3	8145.49	1.46	56.51	9118.40	62.03	285464.50	1.54	1.55E-05	6.73E-03	1.12E+00	7.47E-03	3.50E+01
4	8102.79	1.61	60.88	8811.50	61.57	277110.69	1.84	-3.50E-06	7.27E-03	1.09E+00	7.42E-03	3.42E+01
5	8010.32	1.38	57.89	8597.68	56.89	272361.94	3.69	-2.63E-04	6.75E-03	1.07E+00	6.69E-03	3.40E+01
					Average Bkg:		2.15E+00					
					Average Ratios:			-7.96E-05	7.33E-03	1.14E+00	7.59E-03	3.60E+01
					Std Dev		8.01E-01	1.02E-04	6.57E-04	7.55E-02	5.63E-04	2.36E+00
					%RSD		37.25	-128.03	8.97	6.60	7.43	6.55

	234	235	236	238
TRUE	1.90E-04	3.05E-02	2.00E-04	9.69E-01
MEASURES	1.97E-04	3.08E-02	2.04E-04	9.69E-01
Alpha	1.038	1.009	1.021	1.000

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01B), Procedure (010925b), Element Menu (uiso)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 02/09/02

Analyst: JMBrown 2/25/02
Reviewed by: tmf 2-29-02

Unless otherwise specified; the results are reporte μCi analyte/g of original sample.

RPL Log-In #	Sample ID	ICP/MS ID	MDL μCi/g		Tc 99 μCi/g	±	Tc 99 1SD	Rec/RPD %
	1%HNO_3	ICB		<	7.91E-07			
	1%HNO_3	CCB1		<	7.61E-07			
	1%HNO_3	CCB2		<	8.69E-07			
	True Value				3.39E-06			
	ICV	ICV			3.71E-06	±	9.80E-09	110%
	CCV	CCV1			3.37E-06	±	4.16E-08	99%
	CCV	CCV2			3.38E-06	±	6.23E-08	100%
01-01017-PB	PROCESS BLK	Sample1	3.95E-03	<	3.95E-03	±	5.09E-04	
01-01017-PB	PROCESS BLK post spike	Sample3	3.99E-03		8.85E-03	±	1.74E-04	
Post Spike Concentration expected					8.82E-03			100%
01-01017-BS2	ICP Blank Spike-2	Sample2	3.91E-03	<	3.91E-03	±	8.44E-05	
01-01017	LS-16	Sample4	1.99E-02		7.37E-02	±	3.38E-04	
01-01017	LS-16 replicate	Sample5	2.08E-02		7.24E-02	±	3.96E-04	1.8%
01-01017-DUP	LS-16-DUP	Sample6	2.20E-02		8.68E-02	±	1.40E-03	16.4%
01-01017	LS-15 post spike 6 DW typo.	Sample8	2.25E-02		1.18E-01	±	9.04E-04	
Post Spike Concentration expected					4.41E-02			100%
01-01017-MS2	ICP Matrix Spike-2	Sample7	2.11E-02		8.32E-02	±	8.93E-04	

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 10/1/01
Instrument Filenames: Experiment (01OCT01), Procedure (011001a), Element Menu (Tc)

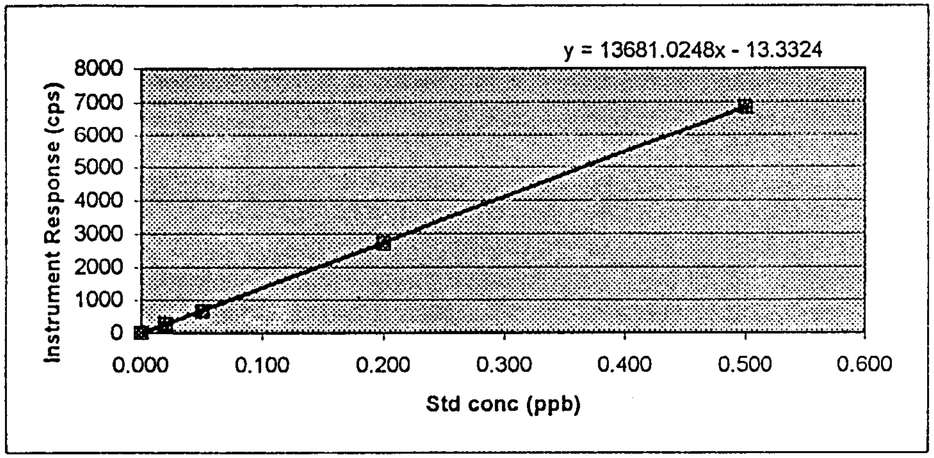
Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/9/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: 2/25/02
Reviewed by: 2-9-02

Technetium - 99

Unless otherwise specified; the results are reported i μCi analyte/g of original sample.



Slope	13681.0248
Intercept	-13.3324
correl	1.0000

Std	
(ppb)	(Cts/sec)
0.000	0
0.020	269
0.050	652
0.200	2715
0.500	6832

RPL Log-In#	Sample ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg acps bkg-sub	Std Dev	Conc. μCi/g	MDL μCi/g		Tc 99 μCi/g	±	1SD
	1%HNO3	ICB	1	1	1	0	1	1.65E-08	7.91E-07	<	7.91E-07	±	3.26E-07
	0.02ppb Tc-99	ICS1	1	1	1	269	4	3.50E-07	8.44E-07	<	8.44E-07	±	1.19E-08
	0.05ppb Tc-99	ICS2	1	1	1	652	13	8.24E-07	8.40E-07	<	8.40E-07	±	1.70E-08
	0.2ppb Tc-99	ICS3	1	1	1	2715	25	3.38E-06	8.10E-07		3.38E-06	±	3.15E-08
	0.5ppb Tc-99	ICS4	1	1	1	6832	66	8.48E-06	8.14E-07		8.48E-06	±	8.20E-08
	0.2ppb Tc-99	ICV	1	1	1	2984	8	3.71E-06	7.59E-07		3.71E-06	±	9.80E-09
	0.2ppb Tc-99	CCV1	1	1	1	2706	33	3.37E-06	7.78E-07		3.37E-06	±	4.16E-08
	1%HNO3	CCB1	1	1	1	3	1	2.04E-08	7.61E-07	<	7.61E-07	±	3.19E-07
01-01017-PB	PROCESS BLK	Sample1	5204	260.2	20	44	6	3.72E-04	3.95E-03	<	3.95E-03	±	5.09E-04
01-01017-BS2	ICP Blank Spike-2	Sample2	5204	260.2	20	204	4	1.40E-03	3.91E-03	<	3.91E-03	±	8.44E-05
01-01017-PB	PROCESS BLK post spike	Sample3	5204	260.2	20	1359	27	8.85E-03	3.99E-03		8.85E-03	±	1.74E-04
01-01017	LS-16	Sample4	26020	260.2	100	2273	10	7.37E-02	1.99E-02		7.37E-02	±	3.38E-04
01-01017	LS-16 replicate	Sample5	26020	260.2	100	2232	12	7.24E-02	2.08E-02		7.24E-02	±	3.96E-04
01-01017-DUP	LS-16-DUP	Sample6	25360	253.6	100	2750	44	8.68E-02	2.20E-02		8.68E-02	±	1.40E-03
01-01017-MS2	ICP Matrix Spike-2	Sample7	24463	244.6	100	2731	29	8.32E-02	2.11E-02		8.32E-02	±	8.93E-04
01-01017	LS-16 post spike	Sample8	26020	260.2	100	3635	28	1.18E-01	2.25E-02		1.18E-01	±	9.04E-04
	0.2ppb Tc-99	CCV2	1	1	1	2716	50	3.38E-06	8.65E-07		3.38E-06	±	6.23E-08
	1%HNO3	CCB2	1	1	1	4	1	2.16E-08	8.69E-07	<	8.69E-07	±	1.65E-07

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 10/1/01
Instrument Filenames: Experiment (01OCT01), Procedure (011001a), Element Menu (Tc)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/9/02

Analyst: [signature] 2/25/0
Reviewed by: [signature] 2-25-02

Unless otherwise specified; the results are reporte μCi analyte/g of original sample.

RPL Log-In #	Sample ID	ICP/MS ID	MDL μCi/g	Np 237 μCi/g	±	1SD	Rec/RPD %
	1%HNO_3	ICB	2.82E-10	6.56E-10			
	1%HNO_3	CCB1	2.78E-10	7.06E-10	±	4.33E-10	
	1%HNO_3	CCB2	3.09E-10	7.70E-10	±	4.69E-10	
	True Value			3.52E-07			
	ICV	ICV		3.48E-07	±	2.38E-09	99%
	CCV1	CCV1		3.49E-07	±	2.89E-09	99%
	CCV2	CCV2		3.52E-07	±	1.94E-09	100%
01-01017-PB	PROCESS BLK	Sample1	1.43E-06	3.99E-06	±	2.08E-06	
01-01017-PB	PROCESS BLK post spike	Sample3	1.54E-06	3.83E-04	±	7.63E-06	
Post Spike Concentration expected				3.67E-04			103%
01-01017-BS2	ICP Blank Spike-2	Sample2	1.44E-06	3.53E-06	±	4.71E-06	
01-01017	LS-16	Sample4	1.51E-05	2.69E-03	±	4.09E-05	
01-01017	LS-16 replicate	Sample5	1.62E-05	2.67E-03	±	1.14E-05	1.0%
01-01017-DUP	LS-16-DUP	Sample6	1.57E-05	2.73E-03	±	2.45E-05	1.4%
01-01017	LS-16 post spike	Sample8	3.25E-05	1.03E-02	±	1.21E-04	
Post Spike Concentration expected				7.34E-03			104%
01-01017-MS2	ICP Matrix Spike-2	Sample7	1.55E-05	2.58E-03	±	4.01E-05	

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01), Procedure (010925a), Element Menu (UNp)

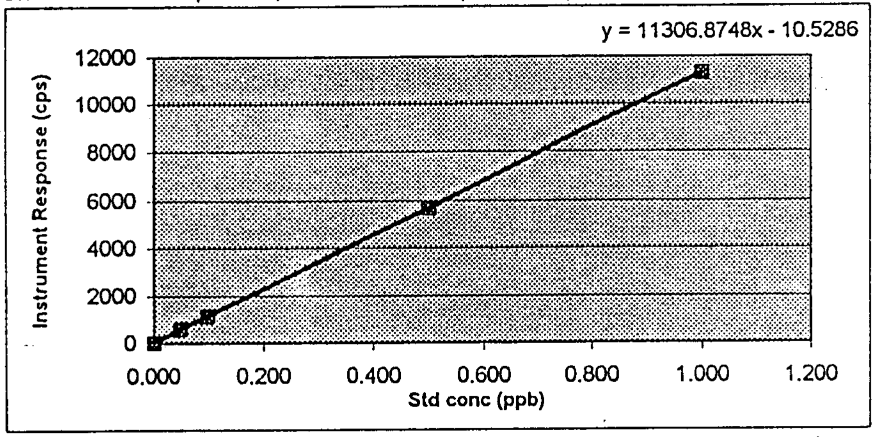
Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/9/02

Analyst: JJBerner 2/25/02
Reviewed by: 4MPO 2-9-02

Neptunium

Unless otherwise specified; the results are reported in μCi analyte/g of original sample.



Slope	11306.8748
Intercept	-10.5286
correl	1.0000

(ppb)	Std (Cts/sec)
0.000	0
0.050	559
0.100	1100
0.500	5651
1.000	11294

RPL Log-In Number	Sample ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg acps bkg-sub	Std Dev	Conc. μCi/g	MDL μCi/g	Np 237 μCi/g	±	1SD
	1%HNO3	ICB	1	1	1	0	1	6.56E-10	2.82E-10	6.56E-10	±	-6.24E-10
	0.05ppb Np	ICS1	1	1	1	559	6	3.55E-08	2.80E-10	3.55E-08	±	3.93E-10
	0.1ppb Np	ICS2	1	1	1	1100	12	6.92E-08	2.77E-10	6.92E-08	±	7.25E-10
	0.5ppb Np	ICS3	1	1	1	5651	6	3.53E-07	3.00E-10	3.53E-07	±	4.06E-10
	1ppb Np	ICS4	1	1	1	11294	11	7.05E-07	2.84E-10	7.05E-07	±	6.75E-10
	10ppb U	ICS5	1	1	1	1	0	7.07E-10	2.59E-10	7.07E-10	±	1.66E-10
	0.5ppb Np	ICV	1	1	1	5578	38	3.48E-07	2.83E-10	3.48E-07	±	2.38E-09
	0.5ppb Np	CCV1	1	1	1	5588	46	3.49E-07	2.75E-10	3.49E-07	±	2.89E-09
	1%HNO3	CCB1	1	1	1	1	0	7.06E-10	2.78E-10	7.06E-10	±	4.33E-10
01-01017-PB	PROCESS BLK	Sample1	5204	260	20	2	1	3.99E-06	1.43E-06	3.99E-06	±	2.08E-06
01-01017-BS2	ICP Blank Spike-2	Sample2	5204	260	20	0	0	3.53E-06	1.44E-06	3.53E-06	±	4.71E-06
01-01017-PB	PROCESS BLK post spike	Sample3	5204	260	20	1171	23	3.83E-04	1.54E-06	3.83E-04	±	7.63E-06
01-01017	LS-16	Sample4	52040	260	200	820	12	2.69E-03	1.51E-05	2.69E-03	±	4.09E-05
01-01017	LS-16 replicate	Sample5	52040	260	200	812	3	2.67E-03	1.62E-05	2.67E-03	±	1.14E-05
01-01017-DUP	LS-16-DUP	Sample6	50720	254	200	854	8	2.73E-03	1.57E-05	2.73E-03	±	2.45E-05
01-01017-MS2	ICP Matrix Spike-2	Sample7	48926	245	200	835	13	2.58E-03	1.55E-05	2.58E-03	±	4.01E-05
01-01017	LS-16 post spike	Sample8	104080	260	400	1577	18	1.03E-02	3.25E-05	1.03E-02	±	1.21E-04
	0.5ppb Np	CCV2	1	1	1	5635	31	3.52E-07	3.12E-10	3.52E-07	±	1.94E-09
	1%HNO3	CCB2	1	1	1	2	1	7.70E-10	3.09E-10	7.70E-10	±	4.69E-10

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/25/01
Instrument Filenames: Experiment (25SEP01), Procedure (010925a), Element Menu (UNp)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/0[illegible]

Reviewed by: [signature] 2/4/02

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01014 to 01-01016
Report Date: 2/9/02

Unless otherwise specified; the results are reported in μCi analyte/g of original sample.

RPL Log-In #	Sample ID	ICP/MS ID	MDL μCi/g	Tc 99 μCi/g	±	1SD	Rec/RPD %
	1%HNO_3	ICB		2.05E-09			
	1%HNO_3	CCB1		2.80E-09			
	1%HNO_3	CCB2		2.35E-09			
	1%HNO_3	CCB3		3.48E-09			
	1%HNO_3	CCB4		3.51E-09			
	True Value			1.70E-06			
	ICV	ICV		1.62E-06	±	3.43E-08	95%
	CCV1	CCV1		1.62E-06	±	4.61E-08	95%
	CCV2	CCV2		1.66E-06	±	4.23E-08	98%
	CCV3	CCV3		1.82E-06	±	1.56E-07	107%
	CCV4	CCV4		1.67E-06	±	3.74E-08	98%
01-01014-PB	PROCESS BLANK	Sample14	7.16E-07	3.19E-06	±	1.69E-07	
01-01014-PB	PROCESS BLANK post spike	Sample15	8.46E-07	4.70E-04	±	9.08E-06	
Post Spike Concentration expected				4.12E-04			113%
01-01014-BS-2	BLANK SPIKE	Sample16	1.66E-06	5.16E-04	±	9.12E-06	
01-01014	LS-12	Sample17	1.50E-05	6.29E-02	±	2.09E-03	
01-01014	LS-12 replicate	Sample18	1.52E-05	6.30E-02	±	4.24E-04	0.2%
01-01014-Dup	LS-12-Dup	Sample19	1.53E-05	6.26E-02	±	1.94E-04	0.3%
01-01015	LS-13	Sample20	1.54E-05	6.03E-02	±	9.02E-04	
01-01015	LS-13 post spike	Sample22	1.56E-05	6.59E-02	±	2.76E-04	
Post Spike Concentration				6.97E-03			80%
01-01015-MS-2	LS-13 ICP/MS MS	Sample23	1.55E-05	6.10E-02	±	7.72E-04	
01-01016	LS-14	Sample21	8.95E-06	1.20E-02	±	3.08E-04	

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/12/01
Instrument Filenames: Experiment (12SEP01), Procedure (010912a), Element Menu (CsTcRb)

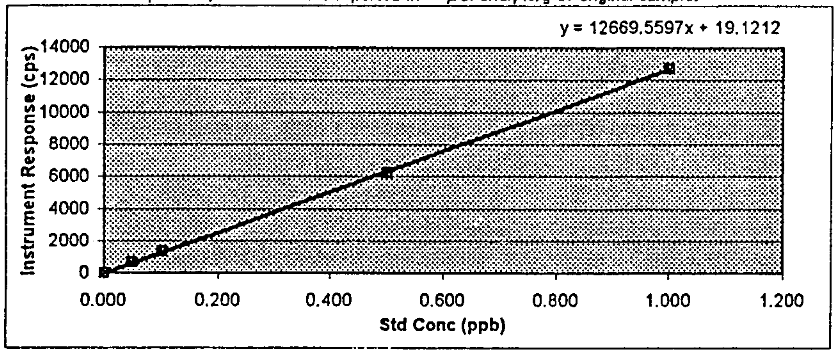
Client: R.Hallen
WP/Project: /W57984 / 42365
ASR/Log-In: 6107, 01-01014 to 01-01016
Report Date: 2/9/02

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [signature] 2/25/
Reviewed by: [initials] 2/9/02

Tc-99

Unless otherwise specified; the results are reported in μCi analyte/g of original sample.



Slope	12669.5597
Intercept	19.1212
correl	0.9999

Std (ppb)	Std (Cts/sec)
0.000	0
0.050	673
0.100	1348
0.500	6242
1.000	12737

RPL Log-In #	Sample ID	Client ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg ecps bkg-sub	Std Dev	Conc. μCi/g	MDL μCi/g		Tc 99 μCi/g	±	1SD
	1%HNO3		ICB	1	1	1	0	0	-2.56E-08	2.05E-09	<	2.05E-09	±	2.12E-09
	0.05ppb Tc-99		ICS1	1	1	1	673	18	8.75E-07	2.27E-09		8.75E-07	±	2.35E-08
	0.1ppb Tc-99		ICS2	1	1	1	1348	12	1.78E-06	2.76E-09		1.78E-06	±	1.58E-08
	0.5ppb Tc-99		ICS3	1	1	1	6242	174	8.33E-06	2.63E-09		8.33E-06	±	2.32E-07
	1ppb Tc-99		ICS4	1	1	1	12737	177	1.70E-05	2.72E-09		1.70E-05	±	2.36E-07
	1%HNO3		w1	1	1	1	231	244	2.84E-07	2.74E-09		2.84E-07	±	2.99E-07
	0.1ppb Tc-99		ICV	1	1	1	1232	26	1.62E-06	2.82E-09		1.62E-06	±	3.43E-08
	0.1ppb Tc-99		CCV1	1	1	1	1228	35	1.62E-06	2.80E-09		1.62E-06	±	4.61E-08
	1%HNO3		w2	1	1	1	9	3	-1.42E-08	2.85E-09	<	2.85E-09	±	1.05E-09
	1%HNO3		CCB1	1	1	1	3	1	-2.22E-08	2.80E-09	<	2.80E-09	±	1.29E-09
	1%HNO3		w3	1	1	1	29	2	1.30E-08	2.27E-09		1.30E-08	±	1.12E-09
	0.1ppb Tc-99		CCV2	1	1	1	1259	32	1.66E-06	2.36E-09		1.66E-06	±	4.23E-08
	1%HNO3		w4	1	1	1	33	19	1.84E-08	2.33E-09		1.84E-08	±	1.05E-08
	1%HNO3		CCB2	1	1	1	8	3	-1.46E-08	2.35E-09	<	2.35E-09	±	7.92E-10
01-01014-PB	PROCESS BLANK	PROCESS BLANK	Sample14	243	24.29	10	29	2	3.19E-06	7.16E-07		3.19E-06	±	1.69E-07
01-01014-PB	PROCESS BLANK post spike	PROCESS BLANK	Sample15	243	24.29	10	1465	28	4.70E-04	8.46E-07		4.70E-04	±	9.08E-06
01-01014-BS-2	BLANK SPIKE	BLANK SPIKE	Sample16	243	24.29	10	812	14	2.58E-04	8.29E-07		2.58E-04	±	4.56E-06
01-01014	LS-12	LS-12	Sample17	4136	20.68	200	11377	378	6.29E-02	1.50E-05		6.29E-02	±	2.09E-03
01-01014	LS-12 replicate	LS-12	Sample18	4136	20.68	200	11395	77	6.30E-02	1.52E-05		6.30E-02	±	4.24E-04
01-01014-Dup	LS-12-Dup	LS-12-Dup	Sample19	4158	20.79	200	11280	35	6.26E-02	1.53E-05		6.26E-02	±	1.94E-04
	1%HNO3		w5	1	1	1	130	132	1.49E-07	3.45E-09		1.49E-07	±	1.51E-07
	0.1ppb Tc-99		CCV3	1	1	1	1379	119	1.82E-06	3.48E-09		1.82E-06	±	1.56E-07
	1%HNO3		w6	1	1	1	34	16	2.01E-08	3.57E-09		2.01E-08	±	9.46E-09
	1%HNO3		CCB3	1	1	1	8	1	-1.50E-08	3.48E-09	<	3.48E-09	±	4.13E-10
01-01015	LS-13	LS-13	Sample20	4111	20.55	200	10987	164	6.03E-02	1.54E-05		6.03E-02	±	9.02E-04
01-01016	LS-14	LS-14	Sample21	2429	24.29	100	3713	95	1.20E-02	8.95E-06		1.20E-02	±	3.08E-04
01-01015	LS-13 post spike	LS-13	Sample22	4111	20.55	200	11998	50	6.59E-02	1.56E-05		6.59E-02	±	2.76E-04
01-01015-MS-2	LS-13 ICP/MS MS	LS-13 ICP/MS MS	Sample23	4044	20.22	200	11287	143	6.10E-02	1.55E-05		6.10E-02	±	7.72E-04
	1%HNO3		w7	1	1	1	132	117	1.51E-07	3.61E-09		1.51E-07	±	1.34E-07
	0.1ppb Tc-99		CCV4	1	1	1	1268	28	1.67E-06	3.66E-09		1.67E-06	±	3.74E-08
	1%HNO3		w8	1	1	1	26	15	8.66E-09	3.60E-09		8.66E-09	±	5.19E-09
	1%HNO3		CCB4	1	1	1	18	3	-8.99E-10	3.51E-09	<	3.51E-09	±	6.64E-10

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/12/01
Instrument Filenames: Experiment (12SEP01), Procedure (010912a), Element Menu (CsTcRb)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/09/02

Analyst: JPBrown 2/25/02
Reviewed by: mPJ 2-9-02

Unless otherwise specified; the results are reported in μCi analyte/g of original sample.

RPL Log-In #	Sample ID	ICP/MS ID	MDL μCi/g	Pu-239 μCi/g	±	1SD	Rec/RPD %	MDL μCi/g	Pu-240 μCi/g	±	1SD	Rec/RPD %
	Eluent Blank (100mM Oxalic Acid/1%HNO_3)	Eluentblk		< 4.46E-11					< 4.21E-11			
	Procedure Blank	Procedureblk	8.11E-11	2.08E-08	±	1.41E-08			< 7.64E-11			
	100mM Oxalic Acid/1%HNO_3	CCB1	5.63E-11	1.55E-07	±	1.16E-08		5.30E-11	9.13E-09	±	1.52E-08	
	100mM Oxalic Acid/1%HNO_3	CCB2	6.00E-11	8.42E-07	±	4.46E-08		5.66E-11	4.64E-08	±	1.97E-08	
	True Value			3.65E-05					2.80E-06			
	ICV	ICV		3.68E-05	±	2.64E-07	101%		2.84E-06	±	6.61E-08	102%
	CCV1	CCV1		3.59E-05	±	2.32E-07	98%		2.77E-06	±	8.38E-08	99%
	True Value			1.22E-04					9.34E-06			
	CCV2	CCV2		1.32E-04	±	4.71E-07	109%		1.03E-05	±	1.09E-07	110%
01-01017-PB	PROCESS BLK	Sample1	1.34E-07	2.98E-05	±	6.05E-06		1.26E-07	4.10E-05	±	2.60E-05	
01-01017-PB	PROCESS BLK post spike	Sample3	1.31E-07	3.86E-02	±	1.04E-03		1.23E-07	2.95E-03	±	4.99E-05	
Post Spike Concentration expected				4.84E-02			80%		3.64E-03			80%
01-01017-BS	ICP Blank Spike-2	Sample2	9.73E-08	2.69E-05	±	9.84E-06		9.17E-08	1.09E-05	±	3.60E-06	
01-01017	LS-16	Sample7	1.06E-07	1.65E-01	±	7.47E-04		1.00E-07	5.77E-02	±	2.93E-04	
01-01017	LS-16 replicate	Sample8	1.21E-07	1.64E-01	±	2.06E-03	0.1%	1.14E-07	5.74E-02	±	7.38E-04	0.6%
01-01017-DUP	LS-16-Dup	Sample4	2.10E-07	1.75E-01	±	9.73E-04	6.2%	1.98E-07	6.05E-02	±	1.22E-03	4.7%
01-01017	LS-16-Dup post spike	Sample6	2.05E-07	2.29E-01	±	2.12E-03		1.93E-07	6.57E-02	±	1.07E-03	
Post Spike Concentration expected				6.29E-02			86%		4.74E-03			109%
01-01017-MS	ICP Matrix Spike-2	Sample5	2.11E-07	1.72E-01	±	1.17E-03		1.98E-07	5.92E-02	±	1.23E-03	

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/26/01
Instrument Filenames: Experiment (26SEP01), Procedure (010926a), Element Menu (Pu)

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: [illegible] 2/25/02

Reviewed by: [illegible] 2.9.02

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/9/02

Pu-239

Unless otherwise specified; the results are reported in μCi analyte/g of original sample.

Pu-242 cts/ppm 10637635.3889

RPL Log-In Number	Sample ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg acps bkg-sub	Std Dev	Conc. μCi/g	MDL μCi/g		Pu-239 μCi/g	±	1SD
	Eluent Blank (100mM Oxalic Acid/1%HNO3	Eluentblk	1	1	1	0	2	0.00E+00	4.46E-11	<	4.46E-11	±	1.63E-11
	Procedure Blank	Procedureblk	1	1	1	4	2	2.08E-08	8.11E-11		2.08E-08	±	1.41E-08
	0.588ppb Pu-239	ICV	1	1	1	6311	45	3.68E-05	8.08E-11		3.68E-05	±	2.64E-07
	0.588ppb Pu-239	CCV1	1	1	1	6152	40	3.59E-05	1.29E-10		3.59E-05	±	2.32E-07
	100mM Oxalic Acid/1%HNO3	CCB1	1	1	1	27	2	1.55E-07	5.63E-11		1.55E-07	±	1.16E-08
01-01017-PB	PROCESS BLK	Sample1	1301	260	5	4	1	2.98E-05	1.34E-07		2.98E-05	±	6.05E-06
01-01017-BS	ICP Blank Spike-2	Sample2	1301	260	5	4	1	2.69E-05	9.73E-08		2.69E-05	±	9.84E-06
01-01017-PB	PROCESS BLK post spike	Sample3	1301	260	5	5089	137	3.86E-02	1.31E-07		3.86E-02	±	1.04E-03
01-01017-DUP	LS-16-Dup	Sample4	1268	254	5	23686	132	1.75E-01	2.10E-07		1.75E-01	±	9.73E-04
01-01017-MS	ICP Matrix Spike-1	Sample5	1223	245	5	24046	164	1.72E-01	2.11E-07		1.72E-01	±	1.17E-03
01-01017	LS-16-Dup post spike	Sample6	1268	254	5	30990	287	2.29E-01	2.05E-07		2.29E-01	±	2.12E-03
01-01017	LS-16	Sample7	1301	260	5	21696	98	1.65E-01	1.06E-07		1.65E-01	±	7.47E-04
01-01017	LS-16 replicate	Sample8	1301	260	5	21679	271	1.64E-01	1.21E-07		1.64E-01	±	2.06E-03
	1.96ppb Pu-239	CCV2	1	1	1	22658	81	1.32E-04	5.21E-11		1.32E-04	±	4.71E-07
	100mM Oxalic Acid/1%HNO3	CCB2	1	1	1	144	8	8.42E-07	6.00E-11		8.42E-07	±	4.46E-08

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/26/01
Instrument Filenames: Experiment (26SEP01), Procedure (010926a), Element Menu (Pu)

Appendix C

Particle Size Distribution

Particle Size Distribution of AN-102 LS15

Hanford Tank 241-AN-102

Adam P Poloski, Author — Date: 3/29/02

Paul R Bredt, Technical Reviewer — Date: 3/29/02

Introduction

The particle size distribution of solids sample from Hanford Tank 241-AN-102 labeled as AN-102 LS15 is described in this report. A Microtrac X-100 Particle Analyzer and a Microtrac Ultrafine Particle Analyzer (UPA) were both used to measure the particle size distribution of the tank samples. The Microtrac X-100 Particle Analyzer measures particle diameter by scattered light from a laser beam projected through a stream of the sample particles diluted in a suspending medium. The amount and direction of light scattered by the particles is measured by an optical detector array and then analyzed to determine the size distribution of the particles. This measurement is limited to particles with diameters between 0.12 and 700 μm. The Microtrac UPA measures particle diameter by Doppler shifted scattered light. This method is limited to particles with diameters between 3 nm and 6.5 μm.

Operating Conditions

The particle size distribution of the AN-102 LS15 sample was measured in the Microtrac X-100 at a flow rate of 40 ml/s. The flow rate was then increased to 60 ml/s and the particle size distribution was measured. The sample was then sonicated with 40W ultrasonic waves for 90 sec at a flow rate of 60 ml/sec and the particle size distribution was measured. Finally, the sample was then sonicated a second time with 40W ultrasonic waves for 90 sec at a flow rate of 60 ml/sec and the particle size was measured. Analyses were performed in triplicate on each sample under all flow/sonication conditions. The average of these triplicate measurements are reported in this document.

For the UPA instrument, no sonication or flow options are available. Therefore, the sample is placed in the instrument and the measurements are performed on the as-received, stationary material. The sample was analyzed in triplicate and the average distribution is reported in this document.

Suspending Medium

The suspending medium for these analyses was a surrogate supernatant based on the tank chemistry of AN-102. A cognizant scientist supplied this suspending medium for use in these particle size instruments.

Calibration Checks

Both instruments performance were checked against a range of NIST traceable standards from Duke Scientific Corporation. These standards are polystyrene microspheres dispersed in a 1 mM KCl solution. These standards were run prior to analysis of the sample. Results from these standard tests are presented in Figures 1 - 4. The percentile data shown in the table represent the given percent of the volume (or mass if the specific gravity for all particles is the same) that is smaller than the indicated particle size. The mean diameter of the volume distribution represents the centroid of the distribution and is weighted in the direction of larger particles. To check the functionality of the instrument, a close fit of the number basis mean data is typically required. The number basis mean results were within 10% of the NIST traceable values.

Figure 1: X-100 Calibration Standards on a Volume Basis (top: differential bottom: cumulative)

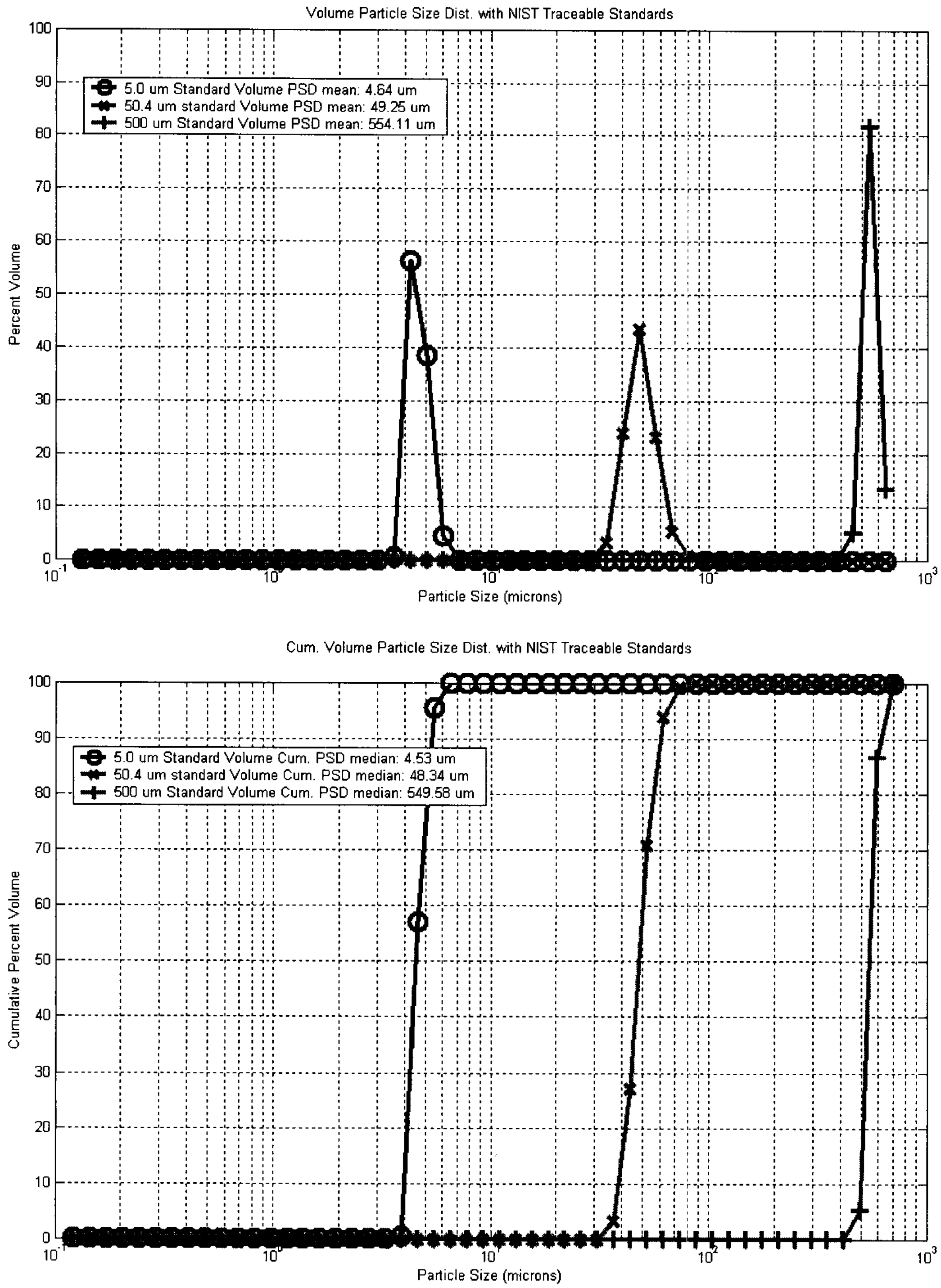
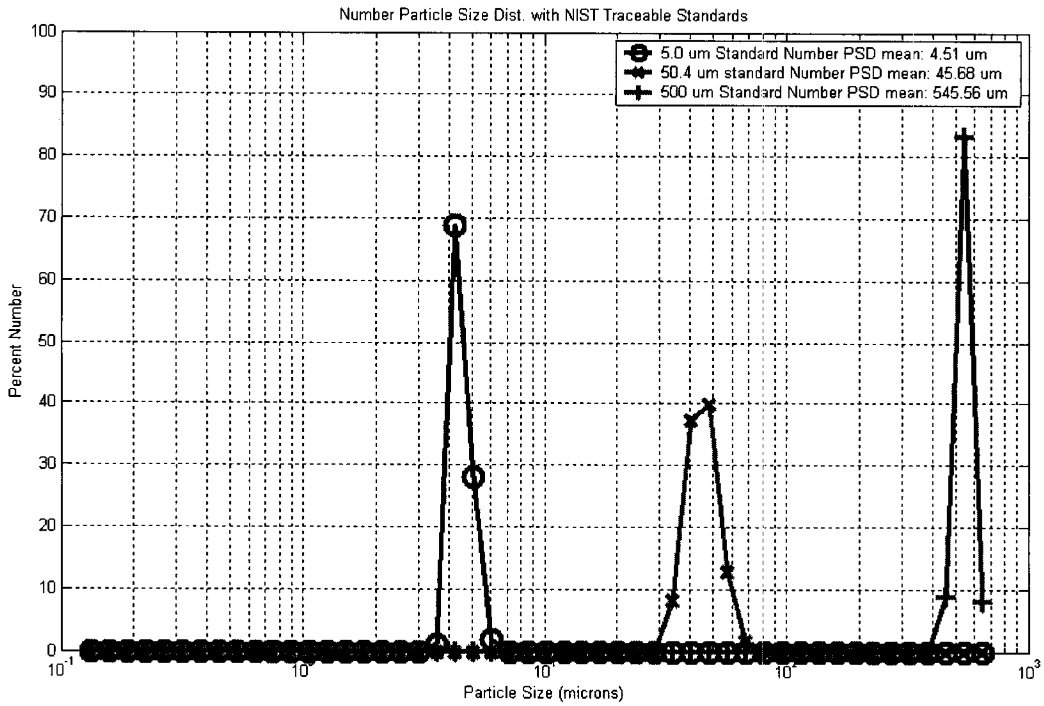


Figure 2: X-100 Calibration Standards on a Number Basis (top: differential bottom: cumulative)



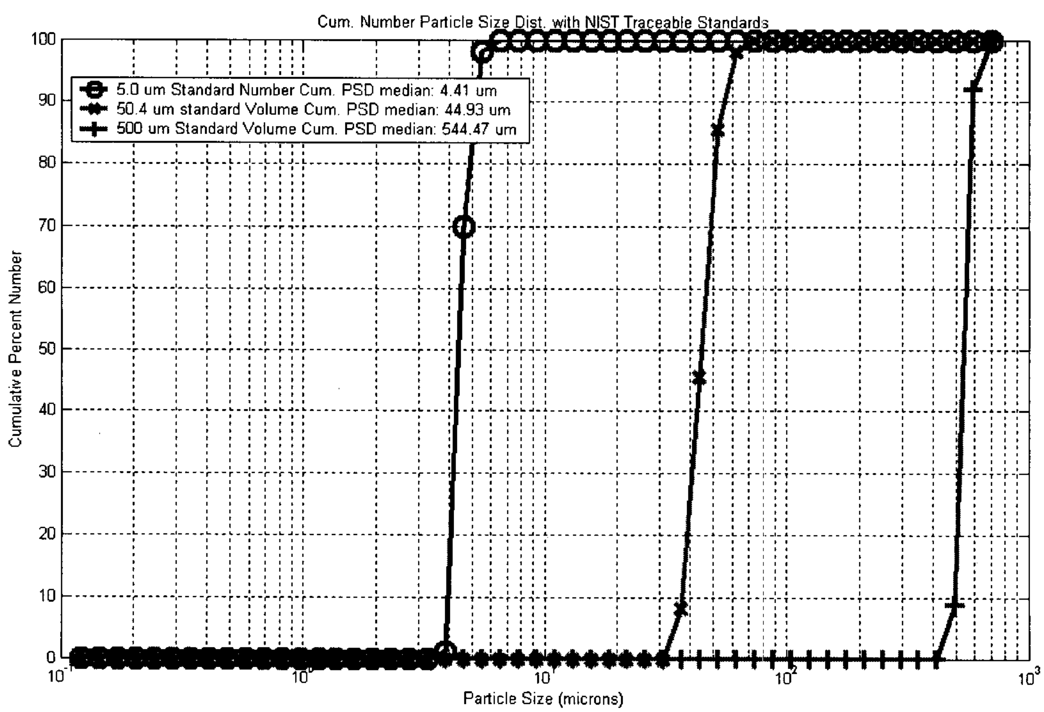
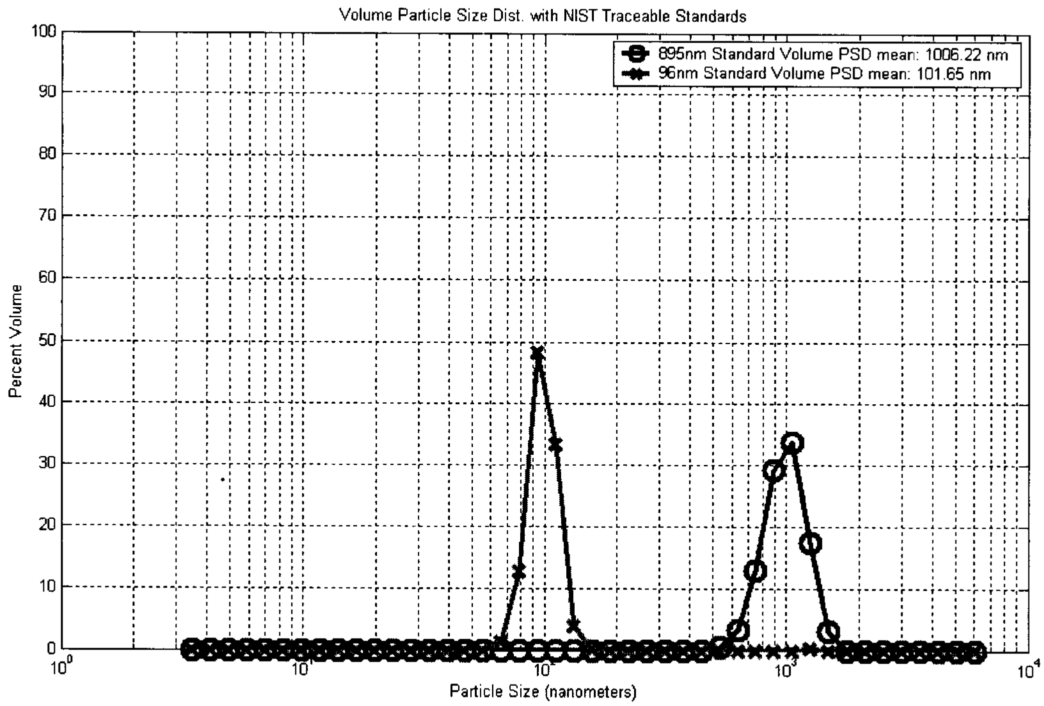


Figure 3. UPA Calibration Standards an a Volume Basis (top: differential bottom: cumulative)



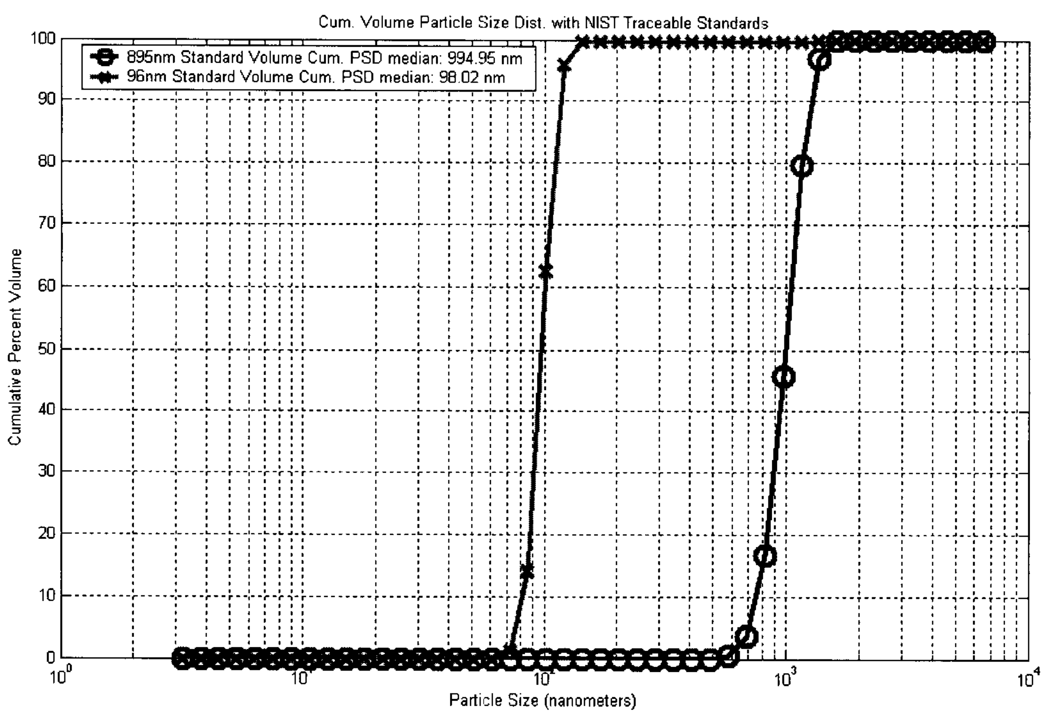
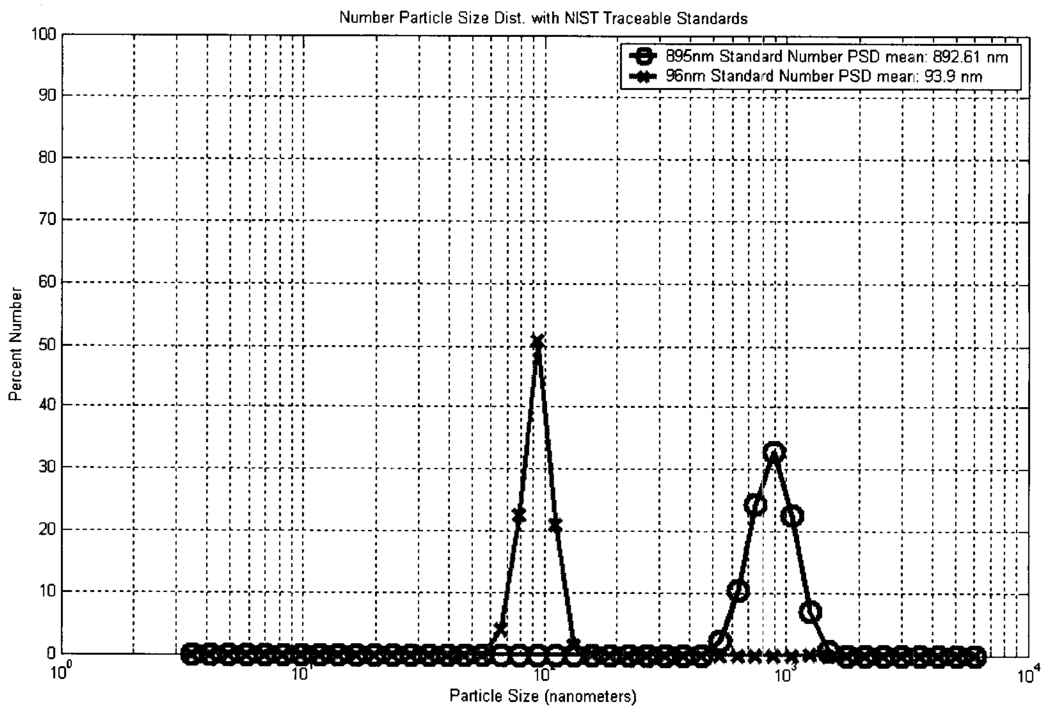
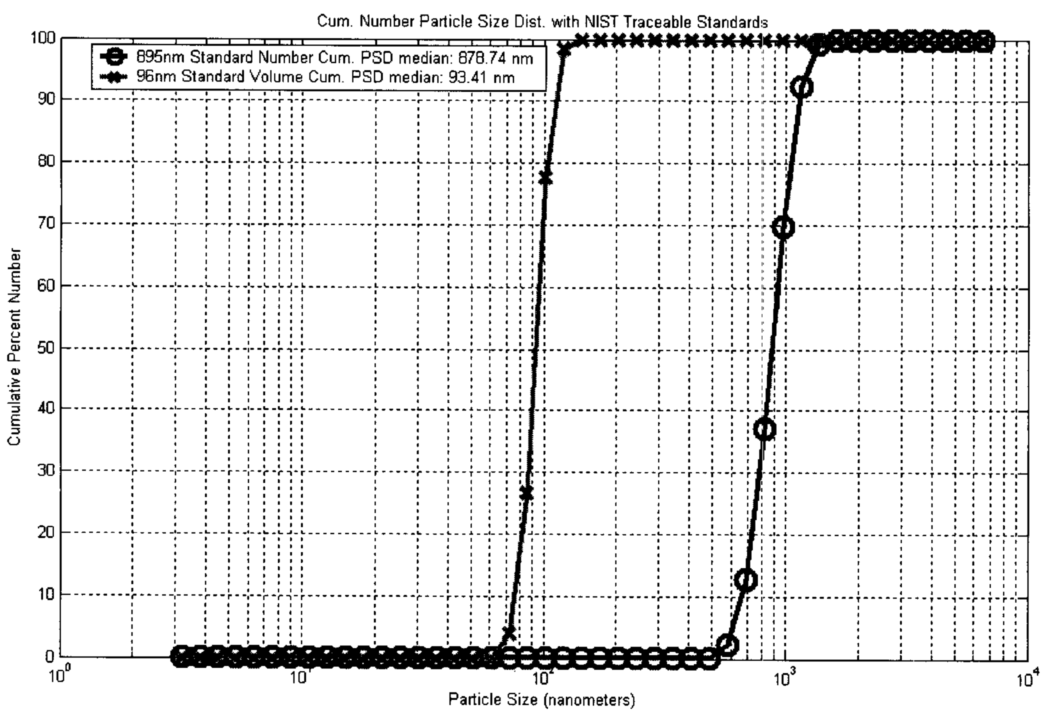


Figure 4. UPA Calibration Standards on a Number Basis (top: differential bottom: cumulative)





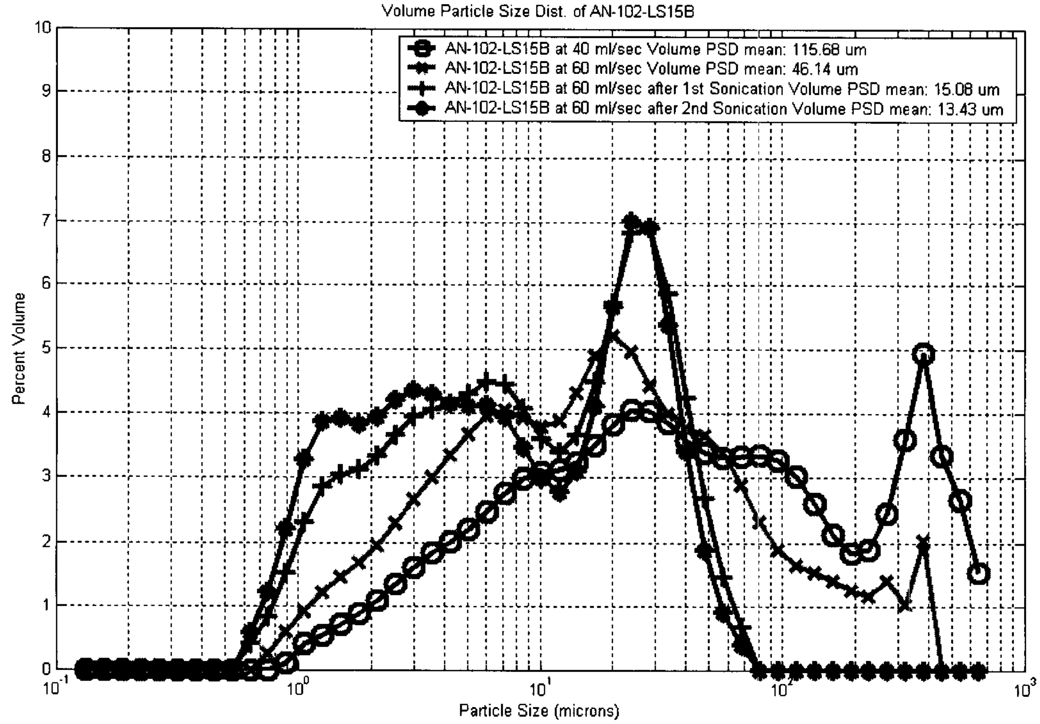
Results

The particle size distributions on a volume basis are presented graphically in Figure 5. In this figure, the top graph presents the volume percent of particles belonging to a discrete range of particle size or bin. The bin-centered values of this histogram are displayed on the graph. The lower graph presents the sum of these data and is referred to as the cumulative volume percent. These data represent the volume percent of particles smaller than a given particle size. For this reason, the right-most bin values are used in this graph. The volume distribution data indicate that most of the slurry volume is due to particles in the 1 to 700 micron particle size range. As the shear rate in the instrument increased (due to increasing the flow rate from 40 mL/sec to 60 mL/sec) some of the larger particles (i.e. particles greater than 80 micron) appear to break apart, resulting in a larger volume of smaller diameter particles. When sonication is applied (i.e. 40W for 90 seconds) at the same flow rate (60 mL/sec), the particles in the 80 to 500 micron size appeared to break apart further resulting in two large peaks at 0.5-10 microns 20-30 microns. A second sonication resulted in a slight amount further deagglomeration.

The particle size distributions on a number basis are presented graphically in Figure 6. Obviously, a large difference in particle size distribution reported between the volume basis and the number basis distributions exist. These differences can be explained by recognizing that as particle size increases the volume contribution of a particle increases cubically. This weighs the volume distribution more heavily towards larger particles (i.e. a few of the larger particles will greatly effect the volume basis but will have no significant effect on the number basis). Hence, Figure 6 suggests that a large number of particles at between approximately 0.5-4.0 microns. There is a small but significant shift to the left of this peak as the particles are sheared. These small changes in the number of larger particles are greatly amplified on a volume basis (see Figure 5).

As-received samples were also analyzed in the UPA. The particle size range that is common to both the X100 and UPA is 0.12 – 6.5 microns. The UPA data is consistent with the X100 data over this range on both a volume basis (see Figure 7). However, on a number basis a large number of particles at approximately 400 nm was measured by the UPA (see Figure 8) and not observed by the X100 (see Figure 6). This might be explained by the greater sensitivity of the UPA instrument at this particle size.

Figure 5. X100 Particle Size Distribution of AN-102 LS15 on a Volume Basis (top: differential bottom: cumulative)



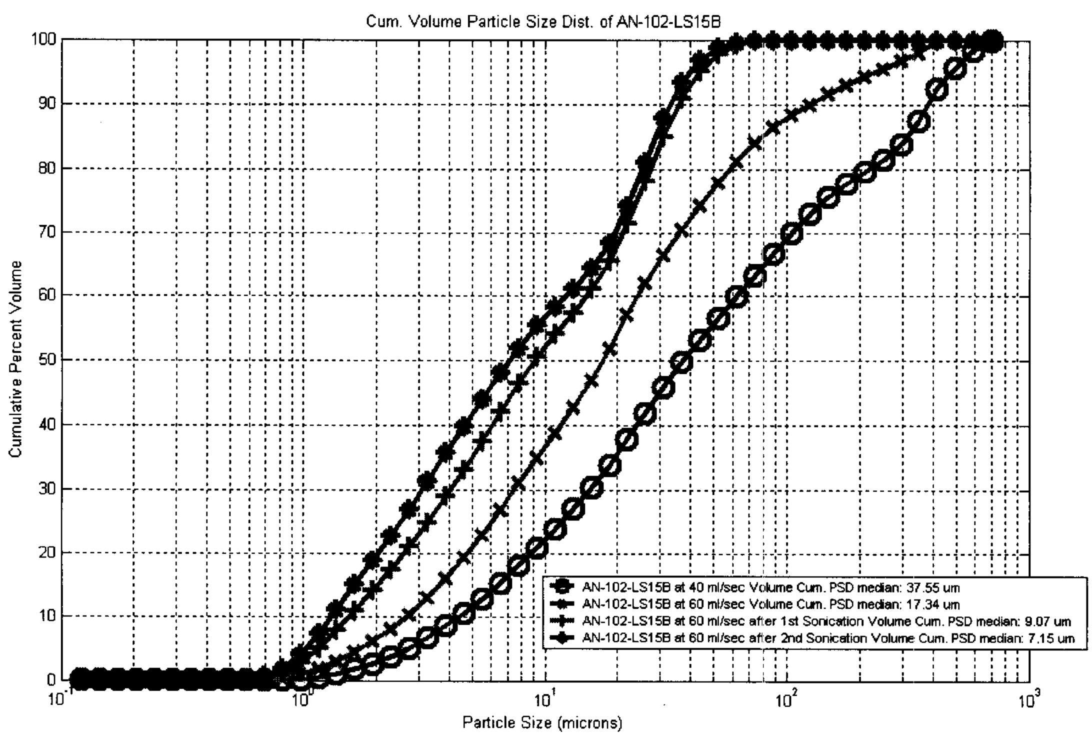
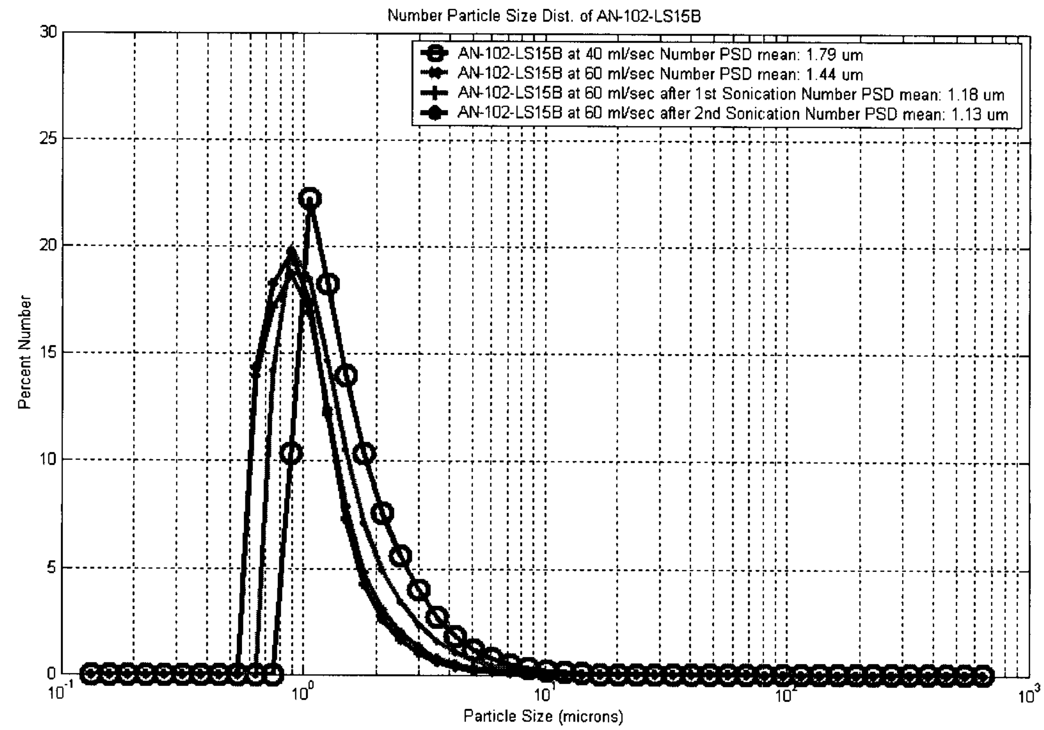
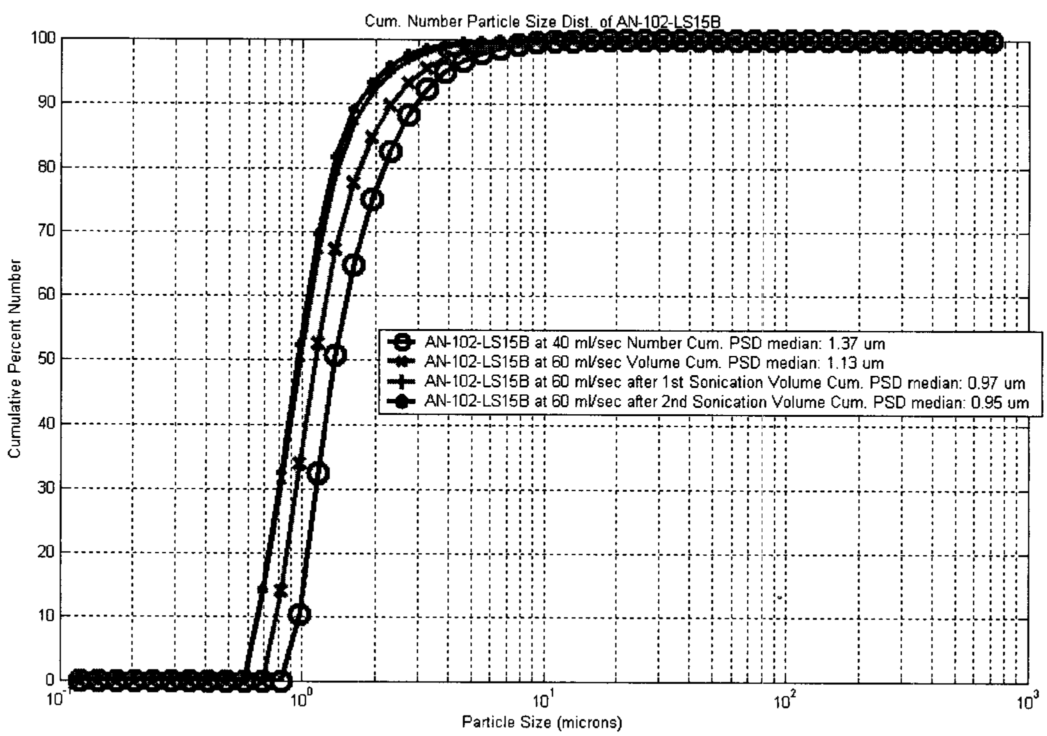


Figure 6. X100 Particle Size Distribution of AN-102 LS15 on a Number Basis (top: differential bottom: cumulative)





Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01017
Report Date: 2/9/02

Analyst: J.L. Bierma 2/25/02
Reviewed By: tmP 2-25-02

Pu-239

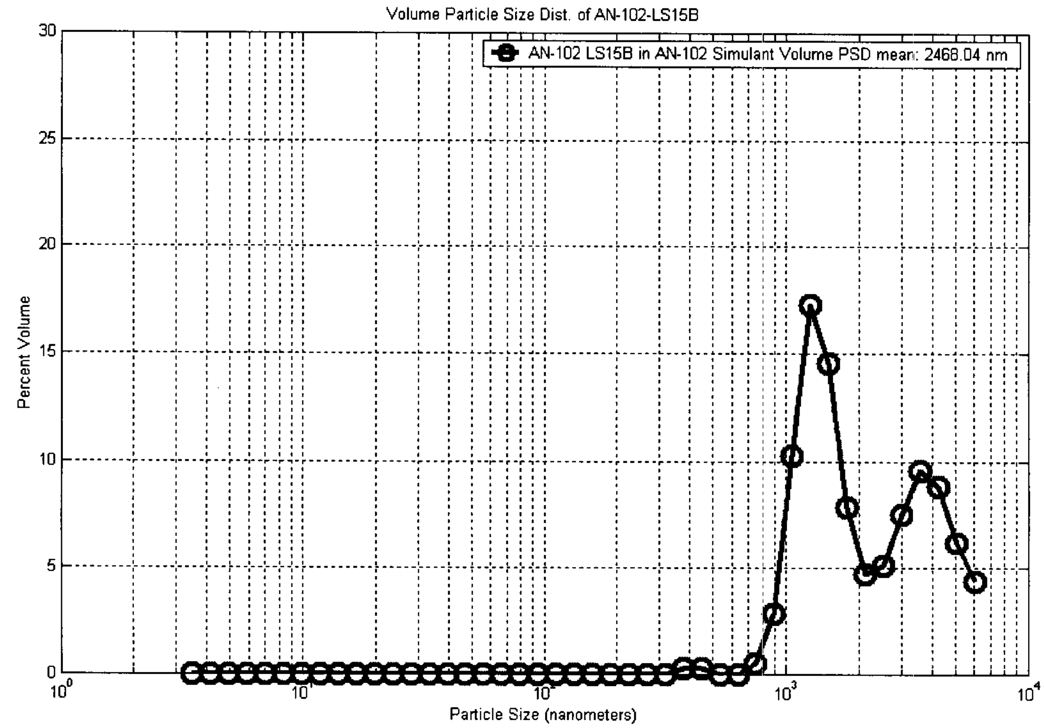
Unless otherwise specified; the results are reported in μCi analyte/g of original sample.

Pu-242 cts/ppm 10637635.3889

RPL Log-In Number	Sample ID	ICP/MS ID	Total Dil.	Client Dil.	ICP/MS Dil.	Avg acps bkg-sub	Std Dev	Conc. μCi/g	MDL μCi/g	Pu-240 μCi/g	±	1SD
	Eluent Blank (100mM Oxalic Acid/1%HNO3	Eluentblk	1	1	1	0	0	0.00E+00	4.21E-11	4.21E-11	±	4.01E-11
	Procedure Blank	Procedureblk	1	1	1	0	0	-5.61E-09	7.64E-11	7.64E-11	±	-1.02E-10
	0.0123ppb Pu-240	ICV	1	1	1	133	3	2.84E-06	7.61E-11	2.84E-06	±	6.61E-08
	0.0123ppb Pu-240	CCV1	1	1	1	130	4	2.77E-06	1.21E-10	2.77E-06	±	8.38E-08
	100mM Oxalic Acid/1%HNO3	CCB1	1	1	1	0	1	9.13E-09	5.30E-11	9.13E-09	±	1.52E-08
01-01017-PB	PROCESS BLK	Sample1	1301	260.2	5	1	1	4.10E-05	1.26E-07	4.10E-05	±	2.60E-05
01-01017-BS	ICP Blank Spike-2	Sample2	1301	260.2	5	0	0	1.09E-05	9.17E-08	1.09E-05	±	3.60E-06
01-01017-PB	PROCESS BLK post spike	Sample3	1301	260.2	5	106	2	2.95E-03	1.23E-07	2.95E-03	±	4.99E-05
01-01017-DUP	LS-16-Dup	Sample4	1268	253.6	5	2237	45	6.05E-02	1.98E-07	6.05E-02	±	1.22E-03
01-01017-MS	ICP Matrix Spike-1	Sample5	1223	244.6	5	2267	47	5.92E-02	1.98E-07	5.92E-02	±	1.23E-03
01-01017	LS-16-Dup post spike	Sample6	1268	253.6	5	2427	40	6.57E-02	1.93E-07	6.57E-02	±	1.07E-03
01-01017	LS-16	Sample7	1301	260.2	5	2079	11	5.77E-02	1.00E-07	5.77E-02	±	2.93E-04
01-01017	LS-16 replicate	Sample8	1301	260.2	5	2066	27	5.74E-02	1.14E-07	5.74E-02	±	7.38E-04
	0.0411ppb Pu-240	CCV2	1	1	1	483	5	1.03E-05	4.91E-11	1.03E-05	±	1.09E-07
	100mM Oxalic Acid/1%HNO3	CCB2	1	1	1	2	1	4.64E-08	5.66E-11	4.64E-08	±	1.97E-08

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/26/01
Instrument Filenames: Experiment (26SEP01), Procedure (010926a), Element Menu (Pu)

Figure 7. UPA Particle Size Distribution of AN-102 LS15 on a Volume Basis (top: differential bottom: cumulative)



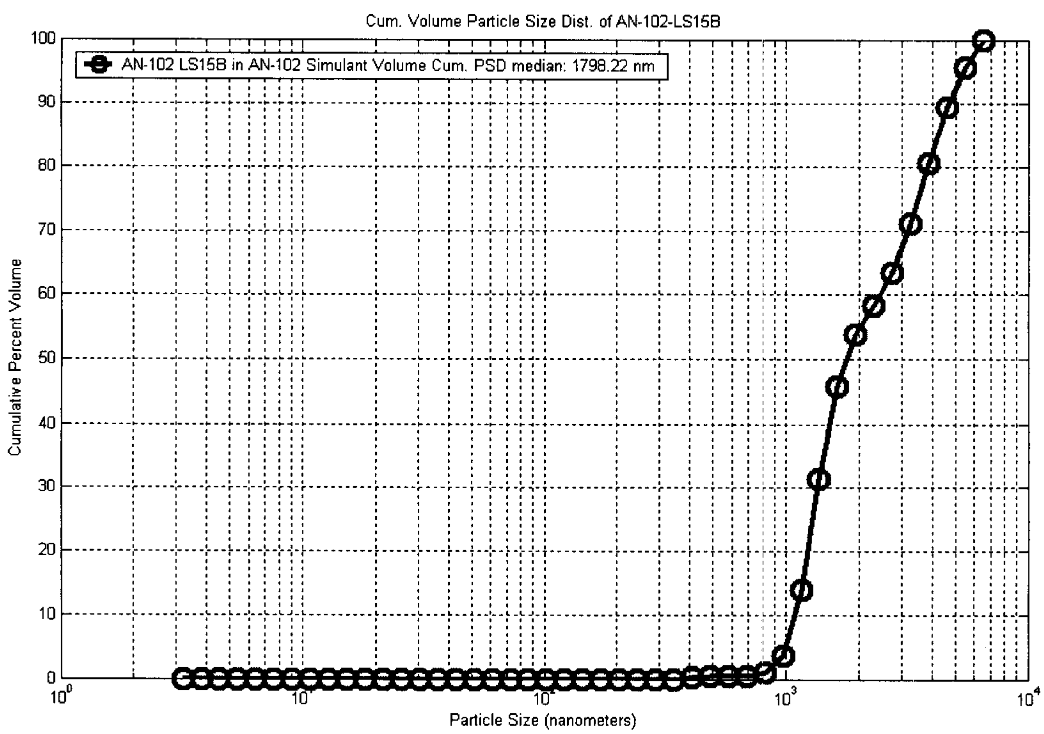


Figure 8. UPA Particle Size Distribution of AN-102 LS15 on a Number Basis (top: differential bottom: cumulative)

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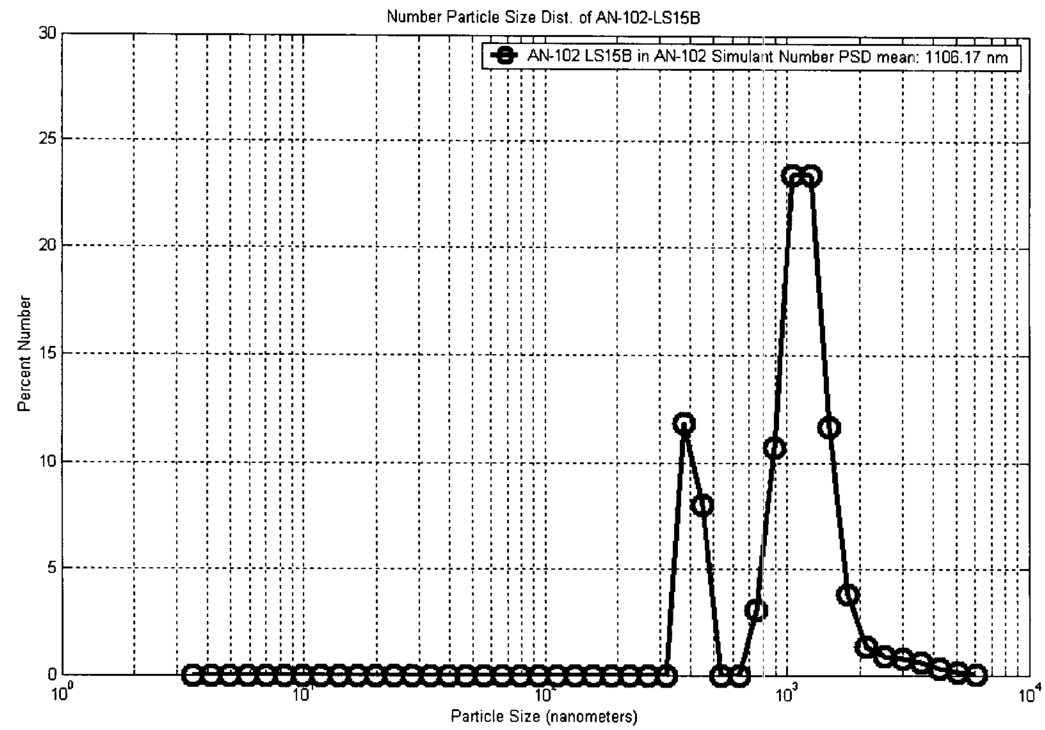

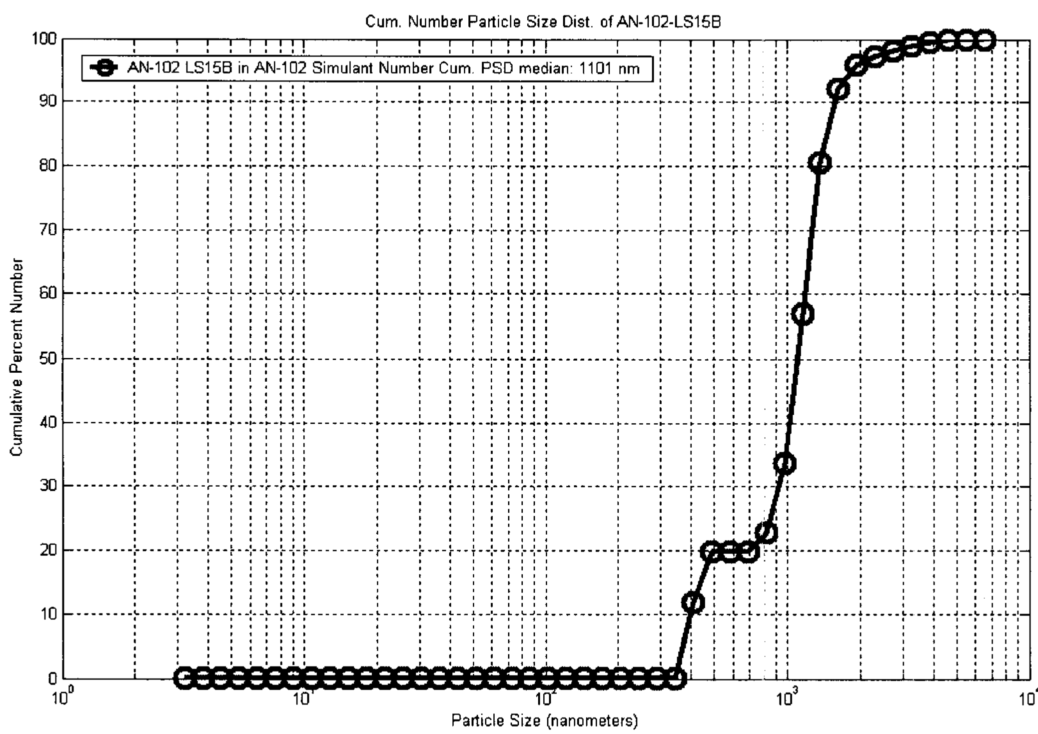

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